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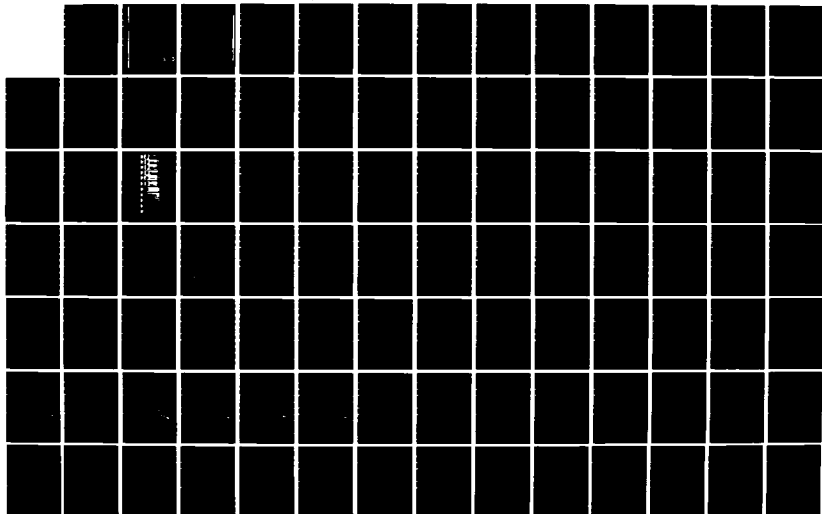
MICROEMULSION-TYPE FIRE-RESISTANT DIESEL FUEL(U)  
SOUTHWEST RESEARCH INST SAN ANTONIO TX ARMY FUELS AND  
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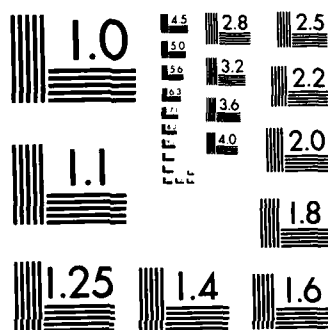
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MICROCOPY RESOLUTION TEST CHART  
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# REPORT ON MICROEMULSION- TYPE FIRE-RESISTANT DIESEL FUEL

INTERIM REPORT  
AFLRL No. 191

By

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Under Contract to

**U.S. Army Belvoir Research  
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19. ABSTRACT (Continue on reverse if necessary and identify by block number) An approach to developing a fire-resistant diesel fuel (FRF) has been identified as being potentially feasible for protecting diesel-powered armored combat vehicles. This approach involves the addition of 10 vol% water and 12 vol% emulsifier premix (equal parts surfactant and aromatic concentrate) to diesel fuel to form a stable water-in-fuel microemulsion. Water, containing less than 50 ppm of dissolved solids, is required if stable formulations are to be made with all diesel fuels which may be encountered. For the same reason, it has been found necessary to include in the formulation an additional 6 vol% of an aromatic hydrocarbon concentrate to serve as a combination microemulsion promotor and emulsifier solvent/thinner. Advanced development of FRF was authorized by a Letter of Agreement on 27 May 1980.  Objectives of continuing FRF-applied research have been to further characterize the current FRF formulation and to address specific critical issues. Results of this				
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research are summarized in this report. The most significant FRF inadequacy is the filter plugging at subfreezing temperatures. This, in turn, precludes FRF use under all weather conditions.

In addition to the foregoing applied research, a parallel program of basic research was conducted in which the influences of changes in emulsifier chemistry on diesel fuel microemulsion phase stability, low-temperature properties, and water purity requirements were studied, and an extensive literature survey was carried out. Overall, none of the modified systems studied produced emulsions that were unambiguously superior to those made by the "standard" FRF surfactant.

## EXECUTIVE SUMMARY

An approach to developing a fire-resistant diesel fuel (FRF) has been identified as being potentially feasible for protecting diesel-powered armored combat vehicles. This approach involves the addition of 10 vol% water and 12 vol% emulsifier premix to diesel fuel to form a stable water-in-fuel microemulsion. The premix contains equal volumes of emulsifier and aromatic concentrate. Although stable FRF formulations can be made with some diesel fuels using water containing more than 500 ppm of total dissolved solids; purified water, containing less than 50 ppm of dissolved solids, is required if stable formulations are to be made with all diesel fuels meeting military specification requirements. For the same reason, it has been found necessary to include in the formulation the additional 6 vol% of an aromatic hydrocarbon concentrate to serve as a combination microemulsion promotor and emulsifier solvent/thinner. Advanced development of FRF was authorized by a Letter of Agreement on 27 May 1980. Objectives of continuing FRF applied research have been to further characterize the current FRF formulation and to address specific critical issues raised in the Letter of Agreement and voiced by potential military users of FRF. Results of this research are summarized in the following paragraphs.

FRF was prepared with over 95 percent of typical diesel fuels if the electrolyte content of the water were 50 ppm or less. Surfactants having a total acid number (TAN) of about 15 were more effective than surfactants with higher acid numbers. Some fuels and water with higher electrolyte content may require a modified surfactant.

Low-temperature flow properties of the FRF compositions were evaluated using equilibrium cooling of the simulated DD 6V-53T engine fuel system. The samples consisted of fuel (No. 11768) blends containing 10, 5, and 2.5 vol% water and surfactant-to-water volume ratio of 6:10. The filter plugging temperature of all samples was approximately -5°C. Flow behavior of FRF was found to be Newtonian with somewhat higher viscosity than the base fuel at temperatures of 0° to 20°C in the low (up to 200) Reynolds number region.

Flammability experiments with FRF-type blends at 66° to 77°C revealed that flame propagation is inhibited across the surface of a fuel pool if water content is between 2.5 and 10 vol%.

Temperature effects on FRF containing excess water were determined by incremental additions of water to the FRF blend at temperatures between 10°C and 30°C. The analysis of the results showed that, at higher temperatures, the FRF of the "standard" base fuel can tolerate up to 16 vol% total water without phase separation. At the lower temperatures, this tolerance is reduced to approximately 12 vol% total water.

Preliminary experiments have indicated the possibility of developing suitable techniques for field monitoring of FRF water content. These include techniques based upon heat of adsorption, coulometric titration, or nuclear magnetic resonance.

Field evaluations of FRF performance in M113 and M60 vehicles were conducted at Yuma Proving Ground and the test results showed that reductions in power were generally less than would be predicted from the lower heat of combustion of FRF, especially with the M60 vehicle. Reduction in the maximum speed of the M113 vehicle was about 11 percent, and only a 2-percent maximum speed reduction was observed with the M60 vehicle. The time for each vehicle to accelerate to 20 mph was increased with FRF by about 3 seconds for the M113 and M60 vehicles.

A parallel program of basic research was conducted in which the influences of changes in emulsifier chemistry on diesel fuel microemulsion phase stability, low-temperature properties, and water purity requirements were studied. Results showed that to reduce sensitivity of aqueous diesel fuel microemulsions to electrolytes in the water, and to extend the fuel's useful temperature range to below 0°C, new or modified surfactants would be needed.



## FOREWORD

This report was prepared at the U.S. Army Fuels and Lubricants Research Laboratory (AFLRL), Southwest Research Institute, San Antonio, TX, under DoD Contract Nos. DAAK70-82-C-0001 and DAAK70-85-C-0007. The project was administered by the Fuels and Lubricants Division, Materials, Fuels, and Lubricants Laboratory, U.S. Army Belvoir Research and Development Center, Fort Belvoir, Virginia 22060, with Mr. F.W. Schaekel, STRBE-VF, serving as Contracting Officer's representative. This report covers the period of performance from 1 January 1982 to 31 December 1984.

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## I. INTRODUCTION

Extensive research has been conducted by the U.S. Army on means for making hydrocarbon fuels resistant to unwanted fire. An approach to developing a fire-resistant diesel fuel (FRF) for use with diesel-powered armored combat vehicles has been identified.(1-6)\* This approach comprises the addition of water and emulsifier to diesel fuel with simple mixing. The resulting FRF is a clear-to-hazy microemulsion. It enhances fuel fire safety by decreasing ignition susceptibility, by retarding flame spread rates, and by self-extinguishing if ignited when spilled. However, it burns readily when atomized, and diesel engines and turbine combustors start, idle, and run satisfactorily on FRF. The viscosity of FRF is somewhat greater than that of its base fuel, especially at low temperatures. It can be pumped at temperatures down to the base fuel pour point; however, it causes filter plugging below 0°C in continuous-flow systems. When burned in an engine, exhaust particulates and flame radiation are diminished, and diesel engine durability is not degraded. FRF is normally stable for more than 30 days, and is not degraded by ambient temperature cycling above 0°C. It can be mixed with other diesel fuels or exposed to normal fuel contaminants without adverse effects on its phase stability. It prevents fuel bacteriological activity and inhibits base fuel deterioration and ferrous alloy corrosion. There are experimental indications that the tendency for the FRF surfactant to corrode copper-containing alloys can be alleviated by the addition of a commercially available additive.

Advanced development of FRF was authorized by a Letter of Agreement (LOA) on 27 May 1980. An executive summary of this LOA is presented in Appendix A. The following approach was established to be the basis for further research following the issuance of the LOA. This was done to accommodate definitions and procedures involved in the development and fielding of a new Army fuel and its production/distribution system. The selected approach involved the addition to diesel fuel of 10 vol% water and 12 vol% emulsifier premix to

---

\*Underscored numbers in parentheses refer to the list of references at the end of this report.



TABLE 12. EFFECT OF ELECTROLYTES ON FRF

Electrolyte, ppm, in Water <sup>a</sup>	Volume %			Rating <sup>e</sup> in Presence of	
	11768 <sup>b</sup>	10716 <sup>c</sup>	EA-96 <sup>d</sup>	NaCl	Ca(NO <sub>3</sub> ) <sub>2</sub>
50	78	6	6	1	1
100	78	6	6	1	1
150	78	6	6	1	1
200	78	6	6	1	2T
300	78	6	6	1T	5
400	78	6	6	5	5
500	78	6	6	5	6 (3%)
1000	78	6	6	6 (4%)	6 (6%)

a. Total water concentration in FRF = 10 vol%

b. Special diesel fuel from Belvoir R&D Center

c. Aromatic concentrate

d. TAN = 19.0 mg KOH/g

e. Visual ratings: (one day after blending)

1. transparent microemulsion
2. translucent microemulsion
3. whitish-brown macroemulsion
4. whitish-yellow macroemulsion
- T. emulsion with trace of cream ( $\leq 0.5$  vol%)
5. microemulsion with less than 2 vol% cream
6. phase separation (vol% lower phase)

The difference between these emulsifiers is the oleic acid content. Emulsifier EA-96 contains significantly more oleic acid (1.7%) than does emulsifier EA-99 as reflected by the differences in their total acid numbers (TAN).

In order to explore the influence of the type of electrolyte on microemulsion phase stability, blends of FRF were made using sodium chloride and calcium nitrate solutions. Table 12 summarizes the findings. It may be seen that both electrolytes impart similar phase destabilizing effects at concentrations above 200 ppm, but the effect of the divalent calcium ion, as expected, is more pronounced than that of the monovalent sodium ion.

Effects of Natural Fuel Deterioration Products on the Phase Stability of FRF--Two fuels known to exhibit poor chemical storage stability were allowed to "age" in clear glass closed vessels on the bench top of an air-conditioned laboratory. Fuel No. 7225 was allowed to age for 10 weeks, while fuel No. 11768 was aged for 6 weeks. Steam jet gum values for the aged, unfiltered fuels were found to be 7.2 and 47.5 mg/100 ml for fuel Nos. 7225 and 11768, respectively. Their respective ASTM D 1500 colors were 3.5 and 6.0. After filtration through millipore filters of 0.8-micrometer porosity, the colors lightened to 3.0 and 5.0, respectively. This filtration removed 14.7 and 82.1 mg of black precipitate from 1000 ml of the aged fuel Nos. 7225 and 11768.

Blends of FRF were made from each of these two base fuels using the fuels in their unaged conditions, as well as after the aging process, before and after filtration. The experimental results are summarized in Table 13. It may be noted that fuel aging had no visually observable deleterious effects on the FRF. An important earlier observation was confirmed in that the FRF did not allow the black precipitate formation that was present in the aged base fuels. This fact may be due either to the antioxidant or dispersant action (or both) of the emulsifying agent.



Table 11. It may be noted that 6 vol% emulsifier, EA-99, is capable of microemulsifying 10 vol% water that contains 50 ppm NaCl, while emulsifier, EA-96, under the same conditions, can microemulsify water containing up to 300 ppm NaCl.

TABLE 11. EFFECTS OF SODIUM CHLORIDE ON FRF

NaCl, ppm in H <sub>2</sub> O <sup>a</sup>	Vol%				Rating <sup>f</sup>
	11768 <sup>b</sup>	10716 <sup>c</sup>	EA-96 <sup>d</sup>	EA-99 <sup>e</sup>	
50	84	-	6	-	1
50	78	6	6	-	1
50	84	-	-	6	1
50	78	6	-	6	2T
100	78	6	6	-	1
150	78	6	6	-	1
200	78	6	6	-	1
300	78	6	6	-	1T
400	78	6	6	-	5
500	78	6	6	-	5
1000	78	6	6	-	6 (4%)
100	78	6	-	6	5
150	78	6	-	6	5
200	78	6	-	6	6 (3%)
300	78	6	-	6	6 (4%)
400	78	6	-	6	6 (4%)
500	78	6	-	6	6 (4%)

a. Total water concentration in FRF = 10 vol%

b. "Standard" base fuel

c. Aromatic concentrate

d. TAN = 19.0 mg KOH/g

e. TAN = 15.5 mg KOH/g

f. Visual ratings:

1. transparent microemulsion
2. translucent microemulsion
3. whitish-brown macroemulsion
4. whitish-yellow macroemulsion
- T. emulsion with trace of cream (<0.5 vol%)
5. microemulsion with less than 2 vol% cream
6. phase separation (vol% lower phase)

TABLE 10. EFFECT OF LIGHT-FUEL CONTAMINATION  
ON FRF

Composition, Vol%								Flash Pt. <sup>i</sup> , °C
11768 <sup>a</sup>	8836 <sup>b</sup>	8511 <sup>c</sup>	9293 <sup>d</sup>	10716 <sup>e</sup>	EA-96 <sup>f</sup>	Water <sup>g</sup>	Rating <sup>h</sup>	
77	1	-	-	6	6	10	1	<27
73	5	-	-	6	6	10	1	<27
77	-	1	-	6	6	10	1	48
73	-	5	-	6	6	10	1	28
77	-	-	1	6	6	10	1	55
73	-	-	5	6	6	10	1	55

a. Base diesel fuel: Flash Point = 56°C

b. Gasoline: RVP = 8.5 lb

c. JP-4: Flash Point = <-29°C

d. JP-8: Flash Point = 43°C

e. Aromatic concentrate

f. TAN = 19.0 mg KOH/g

g. Water contains 50 ppm of NaCl

h. Transparent microemulsion

i. Pensky-Martens closed cup method

TABLE 8. EFFECT OF EXCESS WATER AT 10°C ON FRF\*

Initial Deionized Water Con- tent, Vol%	Phase Distribution, Vol%			Water Content, <sup>1</sup> Vol%			Surfactant Content, <sup>2</sup> Vol%		
	Upper	Middle <sup>3</sup>	Lower	Upper	Middle <sup>3</sup>	Lower	Upper	Middle <sup>3</sup>	Lower
10-12	-----No Phase Separation-----								
12.6	91.7	--	8.3	12.4	--	14.8	5.47	--	6.14
13.5	78.6	--	21.4	12.4	--	17.7	5.4	--	6.33
14.3	78.5	--	21.5	12.8	--	17.2	5.25	--	8.43
15.1	87.2	--	12.8	13.3	--	27.4	4.14	--	7.8
15.9	82.7	--	17.3	13.5	--	27.4	4.98	--	6.42
16.7	2.6	88.2	9.2	12.0	15.7	26.2	4.78	5.47	5.53
17.4	4.2	89.9	5.9	12.8	16.6	22.3	4.46	5.00	5.60
18.2	4.1	90.0	5.9	13.9	17.1	20.1	3.99	5.10	5.38
18.9	2.7	94.1	3.2	16.4	17.5	22.1	4.14	5.07	5.42
19.6	3.0	88.1	8.9	17.2	18.0	23.4	4.65	4.98	5.2

1. By Karl Fischer method

2. By chemiluminescence nitrogen analysis

3. No entry under "middle phase" signifies no middle phase

\* Base fuel: 11768; Emulsifier: EA-90

TABLE 9. EFFECT OF EXCESS WATER AT 0°C ON FRF\*

Initial Deionized Water Con- tent, Vol%	Phase Distribution, Vol%			Water Content, <sup>1</sup> Vol%			Surfactant Content, <sup>2</sup> Vol%		
	Upper	Middle <sup>3</sup>	Lower	Upper	Middle <sup>3</sup>	Lower	Upper	Middle <sup>3</sup>	Lower
10	2	--	98	9.9	--	10.4	6.03	--	5.92
11	6	89	5	4.9	12.0	11.6	3.05	6.18	6.33
12	15	80	5	6.3	12.0	12.9	2.34	6.6	6.18
13	99	--	1	12.9	--	22.7	5.85	--	6.10
14	98	--	2	13.8	--	16.8	5.37	--	6.09
15	96	--	4	14.5	--	26.2	5.44	--	7.45
16	95	--	5	15.8	--	20.6	4.89	--	7.08
17	90	--	10	15.6	--	28.4	5.14	--	7.99
18	2	92	6	6.8	16.0	31.7	4.75	4.84	6.5
19	94	--	6	17.6	--	26.7	4.78	--	7.54
20	87	--	13	18.8	--	26.6	4.49	--	7.00
21	88	--	12	20.6	--	25.5	4.69	--	6.22

1. By Karl Fischer method

2. By chemiluminescence nitrogen analysis

3. No entry under "middle phase" signifies no middle phase

\* Base fuel: 11768; Emulsifier: EA-90

TABLE 6. EFFECT OF EXCESS WATER AT 30°C ON FRF\*

Initial Deionized Water Con- tent, Vol%	Phase Distribution, Vol%			Water Content, <sup>1</sup> Vol%			Surfactant Content, <sup>2</sup> Vol%		
	Upper	Middle <sup>3</sup>	Lower	Upper	Middle <sup>3</sup>	Lower	Upper	Middle <sup>3</sup>	Lower
	-----No Phase Separation-----								
10-16									
17	98	--	2	16.4	--	46.6	5.45	--	**
18	98	--	2	17.6	--	37.1	4.94	--	**
19	2	93	5	--	--	--	4.72	4.81	5.72
20	2	93	5	20.0	19.4	29.9	4.49	4.98	6.08
21	2	93	5	19.6	20.3	30.5	4.44	4.84	6.1

1. By Karl Fischer method

2. By chemiluminescence nitrogen analysis

3. No entry under "middle phase" signifies no middle phase

\* Base fuel: 11768; Emulsifier: EA-90

\*\* Insufficient sample

TABLE 7. EFFECT OF EXCESS WATER AT 23°C ON FRF\*

Initial Deionized Water Con- tent, Vol%	Phase Distribution, Vol%			Water Content, <sup>1</sup> Vol%			Surfactant Content, <sup>2</sup> Vol%		
	Upper	Middle <sup>3</sup>	Lower	Upper	Middle <sup>3</sup>	Lower	Upper	Middle <sup>3</sup>	Lower
	-----No Phase Separation-----								
10-16									
16.7	~100	--	Trace	--	--	--	5.13	--	6.18
17.4	88	--	12	16.9	--	20.4	5.21	--	5.36
18.2	58	--	42	15.9	--	25.6	4.01	--	6.20
18.9	6.4	88.5	5.1	23	18.3	23.1	5.06	5.07	5.79
19.6	14.2	79.9	5.9	19.2	19.4	23.3	4.8	4.44	5.25

1. By Karl Fischer method

2. By chemiluminescence nitrogen analysis

3. No entry under "middle phase" signifies no middle phase

\* Base fuel: 11768; Emulsifier: EA-90

- For 500 ppm or more, the suspension is more stable in base fuel than in base fuel with added emulsifying agent and aromatic concentrate or than in FRF.
- For 500 ppm or more, the suspension is more stable in FRF than in base fuel with added emulsifying agent and aromatic concentrate.

#### Phase Stability:

Temperature Effects on FRF Containing Excess Water--The purpose of this investigation was to determine the amount of additional water an FRF can tolerate as a function of temperature. An original blend of 78 vol% "standard" base fuel (11768) plus 12 vol% emulsifier premix (EA-90) and 10 vol% water (deionized) was subjected to incremental additions of water. Test tubes containing these blends were maintained at 30°, 23°, 10°, and 0°C for 120 hours. The phase volumes, and their component contents are presented in Tables 6-9. Inspection of these data shows that at moderate temperatures of 30° and 23°C, this FRF can tolerate up to 16 vol% total water without phase separation. At 10°C this tolerance is reduced to approximately 12 vol% total water. The FRF (10 vol% total water) itself starts to phase separate at 0°C. When phase separation occurred because of the presence of excess water, the major phase (~80 to 98 volume fraction at all temperatures) had a water content higher than 10 vol% in each case, and, therefore, should continue to be fire resistant.

Effects of FRF Dilution With Other Fuels--Effects of contamination of FRF by gasoline, JP-4 and JP-8 were explored. One to five vol% of these "contaminants" were mixed with base fuel (No. 11768) before addition of 12 vol% of the equivolume mixture of aromatic concentrate (No. 10716) and EA-96, and 10 vol% water that contained 50 ppm NaCl. A summary of these experiments is presented in Table 10. These light hydrocarbon fuels had no visible effects on the microemulsion.

Effects of Water Electrolyte Content--Blends of FRF were made with various sodium chloride concentrations in the emulsified water. Compositional description and evaluation of this experimental matrix are summarized in

TABLE 4. SMOKE POINT OF FRFs AND THEIR COMPONENTS

Composition, Volume Parts						Fuel Comp. TARC, <sup>c</sup> Wt%	Smoke Point, <sup>d</sup> mm
Base Fuel No.			Aromatic Concentrate 10716	Emulsi- fying Agent-99 <sup>a</sup>	Water <sup>b</sup>		
7225	8821	10200					
100	-	-	-	-	-	18.86	14.5
78	-	-	6	6	-	21.48	14.1
78	-	-	6	6	10	21.48	14.8
-	100	-	-	-	-	14.40	15.5
-	78	-	6	6	-	17.33	14.6
-	78	-	6	6	10	17.33	16.0
-	-	100	-	-	-	14.00	17.8
-	-	78	6	6	-	16.99	16.7
-	-	78	6	6	10	16.99	18.0

a. TAN = 15.5 mg KOH/g

b. Water containing 40 ppm of calcium nitrate

c. Total aromatic ring carbon (by UV absorption spectroscopy)

d. ASTM D 1322

TABLE 5. PARTICLE SIZE DISTRIBUTION OF FERRIC OXIDE USED  
(BAKER'S C.P. GRADE)

Equivalent Spherical Diameter, micrometers	Wt%
0.33 to 1	43
1 to 5	12
5 to 10	10
10 to 20	23
20 to 30	9
30 to 50	3
Total	100
2.6	Wt. Median

By: Micromeritics SediGraph 5000D  
Particle Size Analyzer

TABLE 3. COMPARISON OF DISTILLATIONS\* OF AROMATIC CONCENTRATE  
AND BASE FUELS WITH FRF FORMULATIONS BEFORE WATER ADDITION

Vol% Evaporated	Aromatic Concentrate (Code 10716)	78 Vol Parts (7225)				78 Vol Parts (8821)				78 Vol Parts (10200)			
		Base Fuel (Code 7225)	6 Vol Parts Emulsi- fier (EA 99)	6 Vol Parts (10716)	6 Vol Parts (10716)	Base Fuel (Code 8821)	6 Vol Parts Emulsi- fier (EA 99)	6 Vol Parts (10716)	6 Vol Parts (10716)	Base Fuel (Code 10200)	6 Vol Parts Emulsi- fier (EA 99)	6 Vol Parts (10716)	6 Vol Parts (10716)
Distillation Temperature, °C													
IBP	157	166	151	183	160	179	164						
5	160	201	189	211	188	206	189						
10	161	218	201	225	203	218	197						
15	161	222	209	237	219	225	207						
20	161	228	215	247	233	233	215						
30	161	234	225	262	255	245	234						
40	162	238	232	272	268	256	251						
50	162	244	239	282	277	267	265						
60	163	250	247	290	286	279	279						
70	164	257	257	299	296	290	289						
80	167	271	273	312	311	304	303						
90	169	294	295	331	329	324	327						
95	172	314	317	349	343	341	337						
EP	194	358	324	361	345	356	340						
REC.	99.0	98.8	98.5	98.3	98.7	98.5	98.8						
RES.	1.0	1.2	0.9	1.7	1.3	1.5	1.2						
LOSS	0	0	0.6	0	0	0	0						

\* ASTM - D 86

(SPEC22.L)

Boiling point distribution characteristics of FRF ingredients data are presented in Table 3, wherein distillations of an aromatic concentrate (AC) and the base fuels are tabulated for comparison with those of water-free FRF formulations (i.e., FRF before water addition). During the early stages of distillation of the latter mixtures, the effects of the lower-boiling aromatic concentrate are evident. The 90 percent points are essentially unchanged; however, the end points are significantly lower than those of the base fuels. This indicates that the emulsifying agent may have experienced partial decomposition at the higher temperatures.

Smoke points were evaluated for the same three base fuels and their FRF formulations, and the results are listed in Table 4. For each base fuel, the addition of aromatic concentrate increased its smoking tendency, as would be expected, while increasing its aromatic hydrocarbon content. Any effect of the emulsifier was obscured. On the other hand, for each base fuel, the smoking tendency of the water-containing FRF, including the aromatic concentrate, was less than that of the neat base fuel, showing that the added water more than offset the deleterious smoke enhancement effects of the added aromatic hydrocarbons.

Effects of iron oxide particulates on FRF were also investigated with the same three base fuels and their FRF formulations, and the results were inconclusive. The iron oxide was added at eight different concentration levels to each test blend (0-1000 ppm). Table 5 documents the particle size distribution of the iron oxide. On the test blends, ratings taken 24 hours after preparation indicate the following (where stable ferric oxide particle suspensions represent a penalty):

- For 300 ppm or less, the suspension is equally stable in base fuel and base fuel with added emulsifying agent and aromatic concentrate.
- For 300 ppm or less, the suspension is more stable in FRF than in either base fuel or base fuel with added emulsifying agent and aromatic concentrate.



TABLE 2. PROPERTIES OF AN FRF AND ITS BASE FUEL

Property	ASTM D	Base Fuel	FRF <sup>a</sup>
Gravity, °API at 15.5°C	287	32.6	28.0
Density, g/mL at 15.5°C		0.8634	0.8867
Flash point, PMCC, °C	93	56	57
Cloud point, °C	2500	-13	--
Pour point, °C	97	-36	-38
Color	1500	5.5	5.0
Kinematic viscosity, cSt, at 0°C	445	7.56	17.42
Kinematic viscosity, cSt, at 40°C	445	2.62	3.93
Steam jet gum, mg/100 mL	381	30.1	609.0
Accelerated stability, mg/100 mL	2274	23.1	1.1
Copper strip corrosion	130	1A	1A
Total acid number, mg KOH/g	664	0.41	1.22
Heat of combustion, gross, Btu/lb	240	19587	16955
Heat of combustion, gross, MJ/kg	240	45.56	39.44
Heat of combustion, net, Btu/lb	240	18456	15844
Heat of combustion, net, MJ/kg	240	42.93	36.85
Aromatics by HPLC, wt%	-	39.6	--
Aromatic, ring carbon, wt%, by UV			
monocyclic	-	11.83	12.53
dicyclic	-	9.90	7.72
tricyclic	-	0.57	0.42
total	-	22.30	20.67
Water, wt%, by Karl Fischer	1744	0.02	11.2
Sulfur, wt%, by XRF	-	0.14	0.09
Carbon, wt%	3178	87.26	76.58
Hydrogen, wt%	3178	12.40	12.18
Cetane no.	613	42.3	32.1
Distillation, °C, IBP	86 <sup>b</sup>	166	99
5% evap.		201	100
10% evap.		215	100
20% evap.		233	202
30% evap.		247	226
40% evap.		258	246
50% evap.		269	262
60% evap.		279	275
70% evap.		290	288
80% evap.		303	303
90% evap.		323	326
95% evap.		338	342
EP		354	348
Rec.		99.0	99.0
Res.		1.0	1.0
Loss		0.0	0.0

- a. Composition of FRF: Base Fuel No. 11768: 78 vol%  
 Aromatic Concentrate No. 10716: 6 vol%  
 Emsulfier No. EA-96: 6 vol%  
 Water (50 ppm NaCl): 10 vol%

- b. In the case of FRF, the distillation rate for less than 20 percent evaporated was slower than that specified in ASTM D 86.

#### A. Standard Base Fuel

To implement the follow-through FRF research addressing these critical issues, Belvoir R&D Center procured a quantity of commercially available VV-F-800B-DF-2 (AFLRL Code 11768) diesel fuel to serve as a "standard" base fuel. Inspection data for this fuel and the current FRF formulation made with it are presented in Table 2. It is of special interest to note that this dark diesel base fuel has a relatively low flash point and high aromatic hydrocarbon content. It exhibits very low stability values as expressed by its accelerated stability and steam jet gum values. The FRF comprises 78 vol% of the base fuel, 6 vol% of aromatic concentrate, 6 vol% of emulsifier, and 10 vol% water. The following observations may be made when comparing properties of the FRF with those of the base fuel:

- Flash point is essentially unchanged;
- Steam jet gum value of the FRF is very high due to the fact that the emulsifying agent does not completely evaporate under the experimental conditions used in the test;
- Accelerated stability of the FRF is about the same as the specification limit for DF-2 whereas that of the base fuel is drastically worse than that;
- Kinematic viscosity of the FRF is increased above that of the base fuel by about 50 and 130 percent, at 40° and 0°C, respectively;
- Cetane number of the FRF is 10 units lower than that of the base fuel; and
- Net heat of combustion of the FRF is approximately 14 percent (gravimetric) [or 12 percent (volumetric)] less than that of the base fuel.

#### B. Preparation of FRF

##### Preliminary Experiments:

Before the aforementioned "standard" base fuel became available, properties of FRF with additives were evaluated in three base fuels.

TABLE 1. CRITICAL ISSUES

- A. Preparation of FRF
  - 1. Phase Stability
    - a. Dilution with Excess Water
    - b. Dilution with Other Fuels
    - c. Water Purity Effects
    - d. Fuel Cleanliness Effects
    - e. Temperature Effects
  - 2. Quality Assurance Methodology
  - 3. Counter Measures for Off-Specification FRF
  - 4. Preparation of FRF in CONUS Refineries for OCONUS Use
- B. Flammability Characteristics
  - 1. Minimum Water Required for Self-Extinguishment of Fire at Various Temperatures
    - a. Results of Basic Studies
    - b. Results of Simulated and Actual Ballistic Exposures
    - c. Interaction of FRF with Fire Extinguishers
  - 2. Alteration of FRF Flammability Characteristics by Changes in Formulation Ingredients
- C. Flow Characteristics
  - 1. Low-Temperature Flow--Minimum Temperatures
  - 2. Low-Temperature Filtration--Suction Versus Pressure
  - 3. Is FRF Newtonian?
  - 4. Additive Effects
- D. Engine Applications
  - 1. Starting
    - a. Minimum Temperature
    - b. Ignition Quality--Cetane Improvers
    - c. Adequacy of Cetane Number Test for Predicting Combustion Quality
  - 2. Operation
    - a. Full-Scale Vehicle Test
    - b. Significance of Reduced Heating Value of FRF Relative to Qualified Alternate Fuels
    - c. Cetane Improver Effects
    - d. Climatic Window
  - 3. Maintainability and Endurance
    - a. Assessment of FRF Effects
    - b. Effects of Water Washing Lubricant From Cylinder Walls
    - c. Field Test to Detect Phase-Separated FRF
    - d. Lubricity Properties
- E. Types and Arrangements of Filters in Vehicular Fuel Systems

form a stable water-in-fuel microemulsion.(1) The premix contains equal volumes of surfactant and aromatic concentrate. Research was continued to optimize this formulation in terms of the LOA and user requirements. The current formulations have been developed by this research.(4) Although stable FRF formulations can be made with some diesel fuels using water containing more than 500 ppm of total dissolved solids, purified water, containing less than 50 ppm of dissolved solids, is required if stable formulations are to be made with all diesel fuels which may be encountered at a corps class III supply point as specified in the LOA. For the same reason, it has been found necessary to include in the formulation the additional 6 vol% of an aromatic hydrocarbon concentrate to serve as a combination microemulsion promotor and emulsifier solvent/thinner. This is premixed with the emulsifier before FRF blending.

The objectives of the continuing FRF research have been to further characterize the current FRF formulation and to address specific critical issues raised by the potential military users of FRF. This report presents the results of this continued research and summarizes the results of a parallel basic research program on emulsifier chemistry.

## II. CHARACTERIZATION OF FRF

As the continuing FRF research proceeded, several series of briefings were presented by the Fuels and Lubricants Division, Belvoir R&D Center, to potential FRF users within the U.S. Army. As a result of comments and questions arising from these briefings, the U.S. Army Belvoir R&D Center developed a list of critical issues and specified these as priority data requirements for the continuing FRF research. Table 1 lists these critical issues. The FRF research conducted throughout the remainder of the period of performance was directed toward elucidating these critical issues. The results of this research are presented in the following sections of this report.

TABLE 13. EFFECT OF NATURAL FUEL DEGRADATION PRODUCTS ON FRF PHASE STABILITY

Fuel No.	Unaged Fuel	Composition, vol%					Rating <sup>c</sup>
		Aged Fuel		AC		NaCl, ppm in Water	
		Unfiltered	Filtered	10716 <sup>a</sup>	EA-96 <sup>b</sup>	Water	
7225 <sup>d</sup>	78	--	--	6	6	10	1
7225	78	--	--	6	6	10	1
7225	78	--	--	6	6	10	5
7225	--	78	--	6	6	10	1
7225	--	78	--	6	6	10	1
7225	--	78	--	6	6	10	5
7225	--	--	78	6	6	10	1
7225	--	--	78	6	6	10	1
7225	--	--	78	6	6	10	1
7225	--	--	78	6	6	10	5
11768 <sup>e</sup>	78	--	--	6	6	10	1
11768	78	--	--	6	6	10	1
11768	78	--	--	6	6	10	1
11768	78	--	--	6	6	10	1
11768	78	--	--	6	6	10	2T
11768	78	--	--	6	6	10	5
11768	78	--	--	6	6	10	5
11768	78	--	--	6	6	10	6
11768	--	78	--	6	6	10	1
11768	--	78	--	6	6	10	1
11768	--	78	--	6	6	10	2T
11768	--	--	78	6	6	10	1
11768	--	--	78	6	6	10	1
11768	--	--	78	6	6	10	1
11768	--	--	78	6	6	10	2T

a. Aromatic concentrate

b. TAN = 19.0 mg KOH/g

c. Visual ratings: (one day after blending)

1. transparent microemulsion

2. translucent microemulsion

3. whitish-brown macroemulsion

4. whitish-yellow macroemulsion

d. MIL-P-46162 A (MR), Grade II Referee Fuel, aged 10 weeks

e. Special "standard" diesel fuel, aged 6 weeks

T. emulsion with trace of cream ( $\leq 0.5$  vol%)

5. microemulsion with less than 2 vol% cream

6. phase separation (vol% lower phase)

### Quality Assurance Methodology:

After a batch of FRF has been stored, or as it is being blended, observations and/or analyses will establish if the FRF is satisfactory. If the turbidity of the FRF is similar to or less than that of the sample shown in Tube No. 6 of Figure 1, it is considered satisfactory, provided that water content is correct according to blending system meters or actual analysis. In the event that the FRF is not satisfactory, corrective measures may take several routes, depending on the urgency for the fuel and the availability of analytical equipment. Analytical techniques, as shown below, may be used to evaluate the various fuel blends.

Water content of the FRF may be measured by any standard or recommended laboratory procedures, such as:

1. Karl Fischer titration,
2. Gas chromatographic analysis,
3. Density measurement by hydrometer,
4. Near infrared analysis

No research has been conducted on the field use of any of these methods.

Total dissolved solids (TDS) in the water may be easily measured by evaluating its specific conductance with standard instrumentation. Specific conductance varies from approximately 80  $\mu\text{mhos/cm}$  for 50 ppm TDS to approximately 700  $\mu\text{mhos/cm}$  for 500 ppm TDS.

The surfactant content of the FRF may be measured by the product's nitrogen content. Since the neat emulsifying agent has a nitrogen content of 5.90 wt%, FRF containing 6 vol% or 6.8 wt% of this agent should have a nitrogen content of 0.40 wt%. This value may be conveniently measured using commercially available analyzers, such as those utilizing chemiluminescence, for the analysis of oxidation products of nitrogen compounds.

Total acid number (TAN) is an important quality control indicator for the surfactant, as it measures its oleic acid concentration. The surfactant of choice has a TAN value of  $15.5 \pm 0.5$  mg KOH/g of sample. Such a product was (SPEC22.A)

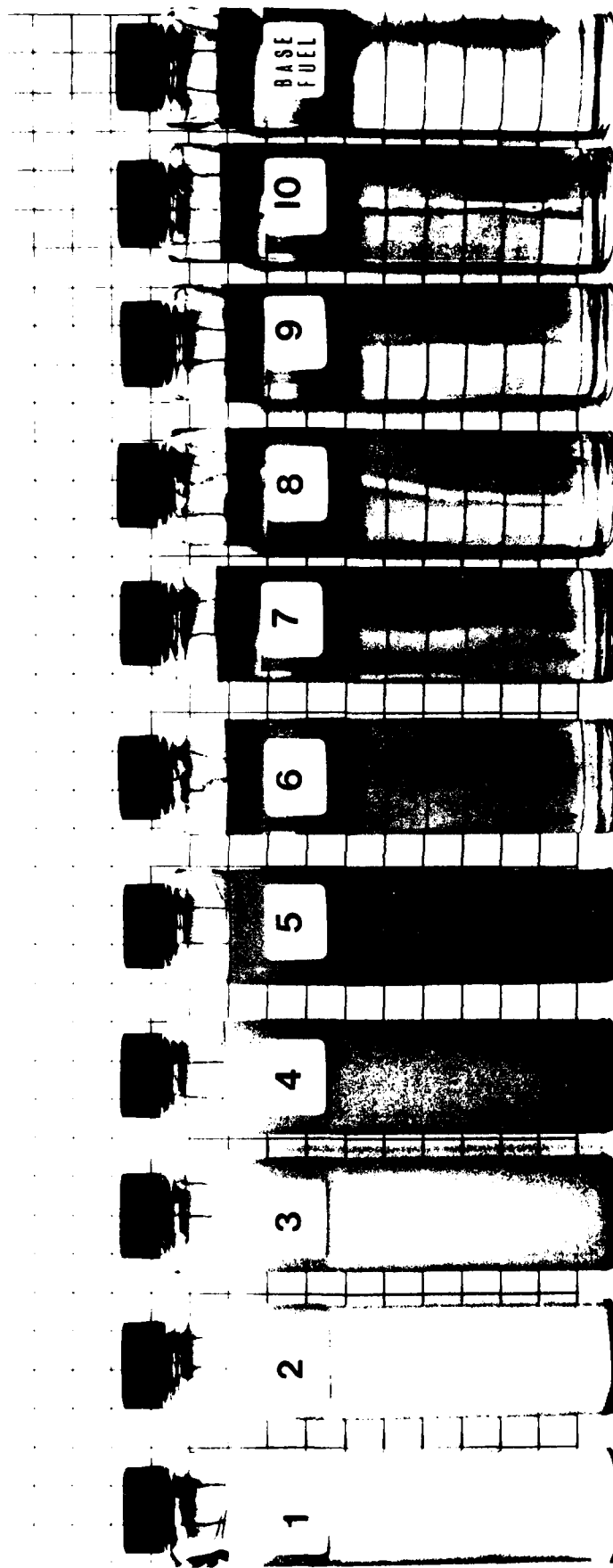


FIGURE 1. PHOTOGRAPH OF A SERIES OF DIESEL FUEL SAMPLES CONTAINING 10 VOL%  
DEIONIZED WATER AND THE INDICATED QUANTITY OF SURFACTANT (1-10 VOL%)  
(Neat Base is Included for Comparison)

shown to emulsify water in over 95 percent of the VV-F-800 and NATO F-54 diesel fuels, provided that the total dissolved solids (TDS) content of the water was 50 PPM or less. Some fuels, and higher TDS concentrations in the water, however, may require the use of modified surfactants, with TAN values of up to 20. It should be emphasized, however, that in comparison with the "standard" surfactant, the modified surfactant will emulsify water in a lower percentage of the available fuels. Measurement of TAN is described in ASTM D 664 and can be modified, for example, by increasing the surfactant's oleic acid concentration by 1.0 wt% will increase its TAN value by 2.

In some cases, the unaltered diesel fuel, or diesel fuel containing only emulsifying agent and aromatic concentrate (i.e., no water), could be used as a vehicular fuel. Naturally, such a product would have no resistance to fire, although it would exhibit strong detergency.

Determination of Water in FRF--A simple gas chromatographic method to measure water content in FRF has been investigated. The initial effort was modestly successful. A 183-cm long x 0.64 cm ID glass column packed with Tenax-GC adsorbent allowed the water to pass into the thermal conductivity detector, while readily adsorbing the fuel and the surfactant. This approach is straightforward and rapid (4 minutes/analysis), but accurate quantitation has not been established, as shown in Table 14.

TABLE 14. GC ANALYSIS OF WATER IN FRF

Sample No.	Composition, Vol%				Water, Vol% Found	Error, %	
	8821 <sup>a</sup>	10716 <sup>b</sup>	EA-99 <sup>c</sup>	Water <sup>d</sup>		Abs.	Rel.
1	79	10	10	1	0.76	0.24	-24
2	74	10	10	6	4.10	1.90	-32
3	70	10	10	10	7.85	2.15	-22
4	87	6	6	1	0.71	0.29	-29
5	82	6	6	6	4.79	1.21	-21
6	78	6	6	10	6.78	3.22	-32
7	100	--	--	--	0.0	--	--

a. Diesel fuel

b. Aromatic concentrate

c. TAN = 19.0 mg KOH/g

d. 50 ppm NaCl solution



Measurement of FRF density by a hydrometer may be used to determine the water content in the fuel blend, provided that the base fuel/aromatic concentrate/surfactant ratio remains constant, and if their individual densities are known. In Table 15, some calculated and measured densities are given.

A brief investigation was conducted to evaluate the possibility of quantifying water-induced deactivation of silica gel. Small disposable pipettes were filled (dry or under heptane) with activated FIA-type silica gel, topped by various water-soluble, fuel-insoluble dyes. Addition of aqueous diesel fuel emulsions to these "chromatography columns" resulted in the ability of differentiating between neat diesel fuel (where the dyes did not discolor the silica gel) and the emulsions (where discoloration of the adsorbent was observed). Higher water content caused higher discoloration of the silica gel. These cursory results indicated that insufficient differences would be generated by this method to afford measurement of water content within acceptable limits. Therefore, no further exploration of the method was conducted.

A number of manufacturers of analytical instruments for chemical laboratories were contacted in search of a simple instrument capable of measuring the water content of FRF under "field conditions" while requiring minimal assistance by an operator.

Several manufacturers claimed that their instruments are rugged, production-type units that could be used "as is," or may be easily adapted to Army use.

1. Digilab of Cambridge, MA, used Fourier-Transform Infrared Spectroscopy (FT-IR) in analyzing two FRF samples that AFLRL sent them. Their results are summarized in Table 16.

2. Moisture Systems of Hopkinton, MA, used a near-infrared analyzer, "Quadra-Beam 575," for the analysis of AFLRL samples. They claim that labor in the instrumental technique is divided into two areas:

TABLE 15. DEPENDENCE OF DENSITY ON FRF WATER CONTENT

No.	7225	8821	10200	10716 <sup>a</sup>	EA-96 <sup>b</sup>	EA-99 <sup>c</sup>	H <sub>2</sub> O <sup>d</sup>	Rating	Calc. <sup>e</sup>	Pycnometer	Hydrometer
1	100	--	--	--	--	--	--	--	--	0.8408	0.8404
2	--	100	--	--	--	--	--	--	--	0.8457	0.8455
3	--	--	100	--	--	--	--	--	--	0.8325	0.8321
4	--	--	--	100	--	--	--	--	--	0.8682	0.8680
5	--	--	--	--	100	--	--	--	--	--	0.9882
6	--	--	--	--	--	100	--	--	--	--	0.9882
7	--	--	--	50	50	--	--	--	--	0.9290	--
8	--	--	--	50	--	50	--	--	--	0.9295	--
9	--	--	--	--	--	--	100	--	--	0.9983	--
10	78	--	--	6	6	--	2	1	0.8556	0.8557	0.8565
11	78	--	--	6	6	--	4	1	0.8586	0.8588	--
12	78	--	--	6	6	--	6	1	0.8616	0.8617	--
13	78	--	--	6	6	--	8	1	0.8642	0.8644	--
14	78	--	--	6	6	--	10	2	0.8670	0.8674	0.8675
15	78	--	--	6	6	--	12	2	0.8696	0.8697	0.8707
16	--	78	--	6	--	6	2	1	0.8598	0.8600	0.8606
17	--	78	--	6	--	6	4	1	0.8627	0.8631	--
18	--	78	--	6	--	6	6	1	0.8656	0.8660	--
19	--	78	--	6	--	6	8	1	0.8682	0.8686	--
20	--	78	--	6	--	6	10	2	0.8708	0.8712	0.8713
21	--	78	--	6	--	6	12	3	0.8734	0.8738	0.8745
22	--	--	78	6	6	6	2	1	0.8486	0.8488	0.8488
23	--	--	78	6	6	6	4	1	0.8517	0.8420	--
24	--	--	78	6	--	6	6	1	0.8549	0.8552	--
25	--	--	78	6	--	6	8	1	0.8577	0.8579	--
26	--	--	78	6	--	6	10	1	0.8606	0.8608	0.8611
27	--	--	78	6	--	6	12	2	0.8633	0.8635	0.8638

a. Aromatic concentrate

b. TAN = 19.0 mg KOH/g

c. TAN = 15.5 mg KOH/g

d. 50 ppm NaCl solution

e. Linearly additive on weight fraction basis.

TABLE 16. ANALYSIS OF FRF SAMPLES BY DIGILAB

		Known, Vol%	Found, Vol%	Error, %	
				Abs.	Rel.
Sample A:	Base Fuel, 8821	84.0	78.29	5.7	-6.8
	Surfactant	6.0	5.01	1.0	-16.5
	Water	10.0	16.69	6.7	66.9
Sample B	Base Fuel, 8821	82.0	75.62	6.4	-7.8
	Surfactant	8.0	10.46	2.5	30.8
	Water	10.0	13.93	3.9	39.3

- a. "Periodic calibration once every 180 days" to check zero and span adjustments;
- b. "Routine analyses:"
  - "Suck sample into analyzer and note reading;"
  - "Drain sample."

They calibrated their instrument on two of the AFLRL samples: both were FRF-type samples, one containing 1 vol%; the other 10 vol% of water. Their results are summarized in Table 17.

TABLE 17. ANALYSIS OF FRF SAMPLES BY MOISTURE SYSTEMS

Sample No.	Composition, Vol%				Water, Vol% Found	Error, %	
	8821 <sup>a</sup>	10716 <sup>b</sup>	EA-99 <sup>c</sup>	Water <sup>d</sup>		Abs.	Rel.
1	86	6	6	2	1.35	0.65	-32.5
2	82	6	6	6	5.64	0.36	-6.0
3	80	6	6	8	9.9	1.9	23.8
4	78	6	6	10	10.45	0.45	4.5
5	70	10	10	10	10.02	0.02	0.2

- a. Diesel fuel
- b. Aromatic concentrate
- c. TAN = 15.0 mg KOH/g
- d. 50 ppm NaCl solution

3. "Automatic," normal Karl Fischer titrators are available from a number of manufacturers (Beckman, Brinkman, Fisher, etc.). These units have been installed in several plants for production control; that is, they are supposedly "rugged" and "fool-proof;" their field use is questionable. The automated coulometric titrators are much simpler to use, as they only require the operator to syringe inject a sample of about 10 microliters and push a button. The built-in "computer" gives the answer as "% water." Among others, Photovolt (Indianapolis, IN) and Mitsubishi (Japan) manufactures such units. These instruments appear promising for Army use.

4. A very simple "water meter" was described by D.A. Law\*, in which "water concentration is determined by accurately measuring the small temperature rise in an insulated environment after injecting a fixed amount of test fluid into a mixture of dilution solvent and zeolite adsorbent." The heat of adsorption is detected by a thermistor probe, and the water content is read out directly on the water meter. While this instrument is not produced commercially, the patent rights are reportedly jointly owned by Mobil Oil Corp. and the U.S. Government.

This instrument was evaluated at AFLRL, and the results have shown this procedure to be accurate, simple to conduct, and completely portable.

5. A water-content measurement instrument was developed at the Electrochemical Research Laboratories of the University of Newcastle upon Tyne, England, and is covered by UK Patent Application 27235/78. This instrument is not yet available commercially, but its design is "available for licensing." It is claimed that "in some oils the extent of emulsified water may also be determined up to 15 percent total water content."

FRF Surfactant Requirements--It has been shown that all of the diesel fuels that were investigated could be microemulsified with water if the total acid number (TAN) of the emulsifying agent were correctly set at either 15 or 19

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\* D.A. Law, "The Development and Testing of an Advanced Water-in-Oil Emulsion for Underground Mine Service," Lubrication Engineering, Vol. 37, No. 2, pp. 82-90, 1981.

mg KOH/g, depending upon the fuel's total aromatic ring carbon content (TARC) and the water's total dissolved solids (TDS) concentrations. Adjustment of the surfactant's TAN involves the addition of oleic acid to the surfactant. The added oleic acid reacts essentially instantaneously with the free diethanolamine in the surfactant if intimate contact is assured between the reagents. This action may be hindered under normal conditions by the high viscosity of the components. Originally, this reaction was carried out batch-wide, at slightly elevated temperature (~50°-55°C) to assure lowered viscosity and adequate mixing of the reagents. It was shown that adjustment of the surfactant's TAN value may be made at any of the several stages of FRF preparation. The required amount of oleic acid may be added to the surfactant/aromatic concentrate "premix" (the preferred way), or it may be added to the "premix"/diesel fuel blend, or even to the final blend containing water. The results of these experiments are summarized in Table 18. Both of the selected base fuels are somewhat anomalous as they require the use of "high" TAN value in the emulsifying agent (EA). As results of Table 18 indicate, aqueous microemulsions were prepared from both of these fuels using an equivolume premix of an aromatic concentrate (AC No. 10716) and EA-96 that has a TAN of 19.0 mg KOH/g. Only macroemulsions resulted if EA-99 was used, whose TAN value is 15.5 mg KOH/g. Addition of 0.05, 0.08, 0.10, 0.12, and 0.15 g oleic acid to the mixtures increases the theoretical TAN value of EA-99 from 15.5 to 17.0, 17.9, 18.5, 19.1, and 20.0 mg KOH/g, respectively. It may be seen in Table 18 that microemulsions resulted by the addition of 0.08 g of oleic acid to either the EA+AC premix or to the blend of this premix and the base fuel. When the additional amount of oleic acid was added to the inadequately prepared FRF, the use of almost twice as much oleic acid was indicated. This observation, however, may indicate that the reagents were too dilute to complete the reaction within the time allowed.

There are several implications of the described observations. Custom adjustment of TAN of the surfactant to accommodate various fuels and increased TDS in the water may be done. Instead of possibly needing an inventory of two surfactants with "low" (about 15) and "high" (about 19) TAN values, only a low TAN product would be necessary, together with small quantities of oleic acid as an "additive". A procedure could be worked out to determine the amount of oleic acid additive needed in the FRF blend.

TABLE 18. PHASE STABILITY RESPONSE TO ADDED OLEIC ACID

Cond <sup>a</sup>	Composition, Vol. Parts						OA <sup>h</sup> ,g	Rating <sup>i</sup>
	10135 <sup>b</sup>	11768 <sup>c</sup>	10716 <sup>d</sup>	EA-96 <sup>e</sup>	EA-99 <sup>f</sup>	Water <sup>g</sup>		
A	78	--	6	6	--	10	--	1
A	78	--	6	--	6	10	--	3T
A	78	--	6	--	6	10	0.05	2T
A	78	--	6	--	6	10	0.08	1
A	78	--	6	--	6	10	0.10	1
A	78	--	6	--	6	10	0.12	1
A	78	--	6	--	6	10	0.15	1
B	78	--	6	6	--	10	--	1
B	78	--	6	--	6	10	--	3T
B	78	--	6	--	6	10	0.05	2T
B	78	--	6	--	6	10	0.08	1
B	78	--	6	--	6	10	0.10	1
B	78	--	6	--	6	10	0.12	1
B	78	--	6	--	6	10	0.15	1
C	78	--	6	6	--	10	--	1
C	78	--	6	--	6	10	--	3T
C	78	--	6	--	6	10	0.05	3T
C	78	--	6	--	6	10	0.08	2T
C	78	--	6	--	6	10	0.10	1
C	78	--	6	--	6	10	0.12	1
C	78	--	6	--	6	10	0.15	1
A	--	78	6	6	--	10	--	1
A	--	78	6	--	6	10	--	3T
A	--	78	6	--	6	10	0.05	1T
A	--	78	6	--	6	10	0.08	1
A	--	78	6	--	6	10	0.10	1
A	--	78	6	--	6	10	0.12	1
A	--	78	6	--	6	10	0.15	1
B	--	78	6	6	--	10	--	1
B	--	78	6	--	6	10	--	3T
B	--	78	6	--	6	10	0.05	2T
B	--	78	6	--	6	10	0.08	1
B	--	78	6	--	6	10	0.10	1
B	--	78	6	--	6	10	0.12	1
B	--	78	6	--	6	10	0.15	1
C	--	78	6	6	--	10	--	1
C	--	78	6	--	6	10	--	3T
C	--	78	6	--	6	10	0.05	3T
C	--	78	6	--	6	10	0.08	3T
C	--	78	6	--	6	10	0.10	2T
C	--	78	6	--	6	10	0.12	2
C	--	78	6	--	6	10	0.15	1

a. Condition A: Oleic acid blended with EA + AC premix

B: Oleic acid blended with mixture of EA+AC+base fuel

C: Oleic acid blended with preformed inadequate FRF  
(rating = 6)

b. Base fuel, DF-2

c. Base fuel, "standard" DF-2

d. Aromatic concentrate (AC)

e. TAN = 19.0 mg KOH/g

f. TAN = 15.5 mg KOH/g

g. 50 ppm NaCl solution

h. Oleic acid, grams

i. 1. Transparent microemulsion

2. Translucent (hazy) microemulsion

3. Brownish macroemulsion

T. Contains cream (&lt;0.5 vol%)

6. Phase separation

### C. Flammability Characteristics

#### Effects of Water Content and Fuel Temperature on Pool Burning:

A series of flammability tests was conducted with FRF-type blends comparing the results of impact dispersion tests with horizontal flame propagation tests\*. The matrix included the effect of fluid temperature and water content on the observed pool burning. The experimental results are presented in Table 19 and Figure 2. These results show that a pool of diesel fuel containing 5 percent water and 3 percent surfactant will not support flame propagation across its surface, even when the pool is heated up to 77°C. It appears that the break point composition between propagation or no-propagation lies between 2.5 and 5 percent water. The base fuel used in these experiments (Code No. 11768; flash point 56°C) exhibited no horizontal flame propagation at 55°C.

On the other hand, the base fuel, and the blend containing 2.5 percent water, both at 55°C, exhibit pool burning on the 77°C impact plate. Under these conditions, the 5 percent water blend does not ignite. At fuel temperatures of 66° and 77°C, the break point between pool ignition and no-ignition lies between 5 and 10 percent water, and the break point between sustained burning and self-extinguishment is between 0 and 2.5 percent water. The time to self-extinguishment at these fuel temperatures is observed to decrease significantly when the water content is increased from 2.5 to 5 vol%.

#### Effect of Aromatic Concentrate on FRF Flammability:

Since the aromatic concentrate may contain flammable ingredients which are more volatile than the base fuel, it might be necessary to specify the maximum content of such constituents in order to assure rapid self-extinguishment of FRF. The most obvious property which is sensitive to the presence of such constituents is the closed cup flash point. A series of experiments

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\* The impact dispersion test and horizontal flame propagation test facilities and procedures are described in References 1 and 4.

TABLE 19. EFFECTS OF WATER CONTENT AND FUEL TEMPERATURE  
ON POOL BURNING OF FRF-TYPE BLENDS

Vol% Deionized Water in Blend (Constant Water/EA Volume Ratio of 1.67)*	Horizontal Flame Channel Test		Impact Dispersion Test		
	Channel and Fuel Temp., °C (°F)	Induction Period Before Flame Departure From Wick, sec	Fuel Temp., °C (°F)	Impact Plate Temp., °C (°F)	Time to Self- Extinguishment of Pool Burning, sec
0	55 (130)	No Flame Propagation	55 (130)	77 (170)	37**
2.5	55 (130)	No Flame Propagation	55 (130)	77 (170)	47***
5.0	55 (130)	No Flame Propagation	55 (130)	77 (170)	No Pool Ignition
10.0	55 (130)	No Flame Propagation	55 (130)	77 (170)	No Pool Ignition
0	66 (150)	20	66 (150)	77 (170)	40**
2.5	66 (150)	984	66 (150)	77 (170)	26
5.0	66 (150)	No Flame Propagation	66 (150)	77 (170)	13
10.0	66 (150)	No Flame Propagation	66 (150)	77 (170)	No Pool Ignition
0	77 (170)	10	77 (170)	77 (170)	38**
2.5	77 (170)	80	77 (170)	77 (170)	25, 38
5.0	77 (170)	No Flame Propagation	77 (170)	77 (170)	15, 23
10.0	77 (170)	No Flame Propagation	77 (170)	77 (170)	No Pool Ignition

\* Balance of blend was "standard" fuel (No. 11768) (flash point = 56°C)  
 \*\* All of fuel consumed (i.e., burnout rather than self-extinguishment)  
 \*\*\* Borderline self-extinguishment (essentially extinguished but reignited)



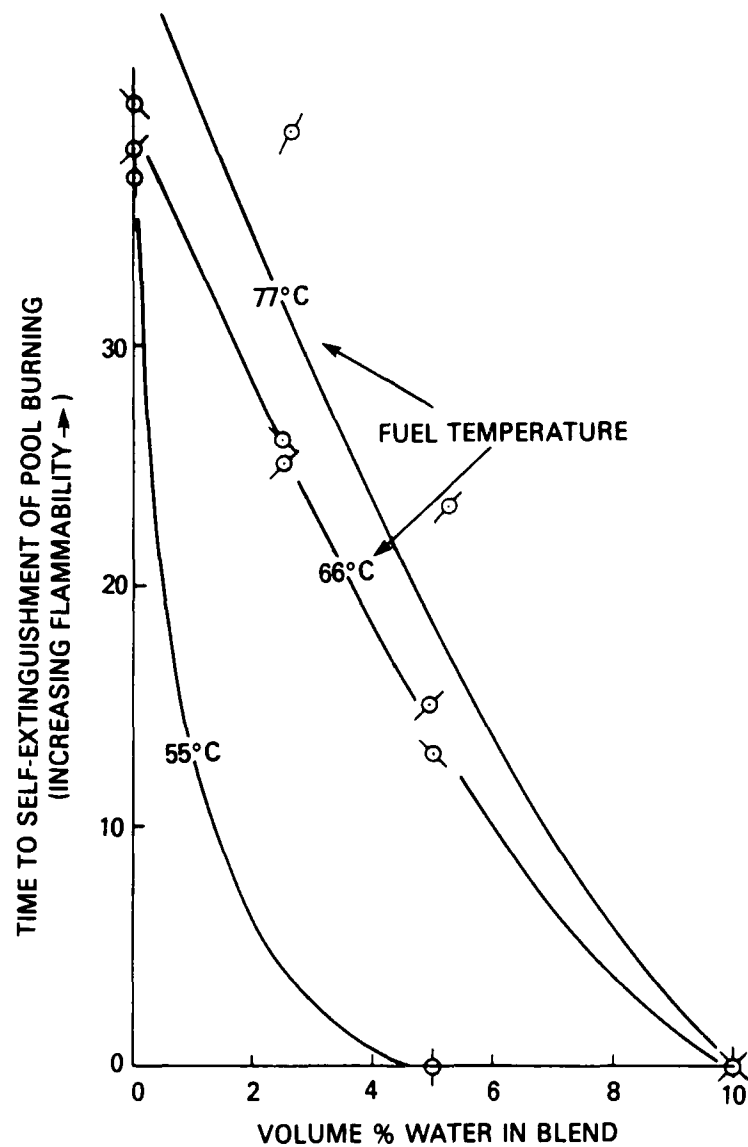


FIGURE 2. INFLUENCES OF WATER CONTENT AND FUEL TEMPERATURE  
ON FLAMMABILITY OF FRF-TYPE BLENDS

was conducted using base fuels and FRF blends containing aromatic concentrates of differing flash point. These tests comprised measurements of flash point and of pool-burning duration in the AFLRL impact dispersion test.(1,4) The latter test has been previously shown to correlate with full-scale and simulated full-scale ballistic tests. The results, which are summarized in Table 20, are portrayed graphically in Figures 3 and 4. In Figure 3, it is apparent that 6 volume parts of aromatic concentrate in 78 volume parts of base fuel exert significant effects on the flash point of the mixture when the flash point of the aromatic concentrate is less than that of the base fuel. The pool-burning duration data shown in Figure 4 indicate that the flash point reductions are accompanied by substantial increases in pool-burning duration for the most volatile base fuel and aromatic concentrate (7725 and 10716, respectively).

#### D. Flow Characteristics

##### Nonturbulent Flow Evaluations:

Evaluation of low-temperature flow and the flow behavior (Newtonian or non-Newtonian) was carried out by measuring pressure drop across a capillary tube at different laminar flow rates and temperatures using the "standard" base fuel (11768).

Figure 5 presents pressure drop vs flow rate data at Reynolds number of 150 at 0°C to 600 at 20°C. These data were collected with a 0.155-cm ID, 152.4-cm long, smooth tubing. The FRF was continuously homogenized with a Kenix-type static mixture before passing through the tubing. Flow rate and corresponding pressure drops were measured at different temperatures. The linearity of data in Figure 5 suggests that the FRF is Newtonian in the region of measurement, and hence the viscosities at different temperature can be calculated from the slopes of the lines. Table 21 presents the viscosities and maximum Reynolds number at the measurement conditions. The range of measurements was limited by the maximum capacity of the mercury manometer (165 kPa) used for accurate measurement of the pressure drop even

TABLE 24. LOW-TEMPERATURE PUMPABILITY: EQUILIBRIUM COOLING--  
DD 6V-53T CONDITIONS--PRESSURED FLOW THROUGH SOCK-TYPE FILTER

<u>FRF Composition:</u>		<u>Vol%</u>
AL-11768-F		78
EA-96		6
AL-10747-A		6
Deionized Water		10

<u>Fuel Temp, °C</u>	<u>Pressures on Sock Filter, psig</u>		<u>Fuel Flow*</u>
	<u>Inlet</u>	<u>Outlet</u>	
25	38	36	Sufficient
-3	56	40	Sufficient
-6	0	0	None

\* Fuel flow: ~125 ml/min, sufficient to start a DD 6V-53T engine.

TABLE 25. LOW-TEMPERATURE FLOWABILITY OF FRF:  
BASED ON AL-11768-F

<u>Temperature, °C</u>	<u>Suction on Fuel Pump, kPa (In. Hg)</u>	<u>Flow*</u>
-5	16.9 (5)	Yes
-10	16.9 (5)	Yes
-13	-----Cloud Point of Base Fuel-----	
-15	20.3 (6)	Yes
-20	27.0 (8)	Yes
-25	30.4 (9)	Yes
-30	33.8 (10)	Yes
-33	37.1 (11)	Yes
-35	47.3 (14)	Yes
-36	-----Pour Point of Base Fuel-----	
-37	54.0 (16)	Yes
-40	54 (16)	Yes
-43	Cavitation	No flow

\* Flow of 1650 ml/min, sufficient to start a DD 6V-53T engine while providing recycle flow.

at 25°, -3°, and -6°C. The sock-type filter plugs under these conditions at approximately -6°C, which is approximately the same temperature at which the no-flow conditions were obtained with the normal filter arrangement.

A series of experiments was conducted in the simulated DD 6V-53T engine fuel system to determine low-temperature flowing ability of the FRF in the absence of any filtering media. The primary and secondary filter cartridges were removed, and the FRF was pumped through the system at different equilibrium temperatures. The suction pressures at the inlet of the fuel pump and the flow conditions as reported in Table 25 show that the FRF can flow adequately at least down to the pour point of the base fuel in the absence of fuel filters (pour point of the base fuel: -36°C).

Effect of Alcohol Cosurfactants on FRF --In an attempt to decrease the minimum temperature for satisfactory operation of FRF in the simulated engine fuel system, several blends were made in which 5 vol% of an alcohol cosurfactant was substituted for 5 vol% base fuel. Three alcohols, secondary-butanol, tertiary-butanol, and n-pentanol, were tried as cosurfactant-type additives in the FRF composition based on fuel (11768). As shown by their ratings in Table 26, none of these alcohols produced a stable microemulsion.

#### E. Engine Applications

##### Engine Starting:

A series of specialized engine tests was conducted with FRF and its ingredients to study the effect of cetane improvers on low-temperature ignition characteristics. These tests were conducted in a Detroit Diesel 4-53T engine that was completely insulated and serviced by a cooling system capable of reaching cold soak temperatures of -20°C. An external cranking motor was used to provide controlled-speed cranking and an inline torque-meter monitored cranking torque. Inlet air was pre-treated by a compressor/air dryer system. The unit was chilled to the desired test temperature, then cranked at 150 rpm for 1 minute. If the engine failed to start after 1

TABLE 23. LOW-TEMPERATURE PUMPABILITY: EQUILIBRIUM COOLING--  
DD 6V-53T CONDITIONS--REDUCED WATER CONTENT FRF-TYPE BLENDS

A:		Composition	Vol%
		AL-11768-F	89
		EA-96	3
		AL-10747-A	3
		Deionized Water	5

Fuel Temp, °C	Pressure on Secondary Fuel Filter, psig		Primary Fuel Filter Pressure Drop, in. Hg	Fuel Flow*
	Inlet	Outlet		
8	20.5	20.0	6	Sufficient
0	21.5	21.0	7	Sufficient
-3	59.5	59.0	8	Intermittent
-5	>60	0	11	None

B:		Composition	Vol%
		AL-11768-F	94.5
		EA-96	1.5
		AL-10747-A	1.5
		Deionized Water	2.5

Fuel Temp, °C	Pressure on Secondary Fuel Filter, psig		Primary Fuel Filter Pressure Drop, in. Hg	Fuel Flow*
	Inlet	Outlet		
10	20.5	20.0	6	Sufficient
0	21.5	21.0	7	Sufficient
-5	0	0	0	None

\* Fuel flow: ~125 ml/min (sufficient to start a DD 6V-53T engine).

TABLE 22. LOW-TEMPERATURE PUMPABILITY: EQUILIBRIUM COOLING--  
DD 6V-53T CONDITIONS--FRF BLENDS

<u>A:</u>	<u>FRF Composition:</u>		<u>Vol%</u>	
	AL-7225-F		84	
	EA-99		6	
	Deionized Water		10	
<u>Fuel Temp,</u> <u>°C</u>	<u>Pressure on Secondary</u> <u>Fuel Filter, psig</u>		<u>Primary Fuel Filter</u> <u>Pressure Drop, in. Hg</u>	<u>Fuel Flow*</u>
	<u>Inlet</u>	<u>Outlet</u>		
0	39	38	6	Sufficient
-2	38	38	7	Sufficient
-4	40	39	8	Sufficient
-5, -6	42	40	8-9	Intermittant
-7	>60	0	>10	None

<u>B:</u>	<u>FRF Composition:</u>		<u>Vol%</u>	
	AL-11768-F		84	
	EA-96		6	
	Deionized Water		10	
<u>Fuel Temp,</u> <u>°C</u>	<u>Pressure on Secondary</u> <u>Fuel Filter, psig</u>		<u>Primary Fuel Filter</u> <u>Pressure Drop, in. Hg</u>	<u>Fuel Flow*</u>
	<u>Inlet</u>	<u>Outlet</u>		
0	45	44	7	Sufficient
-3	47	46	8	Sufficient
-5	15	12	13	None

<u>C:</u>	<u>FRF Composition:</u>		<u>Vol%</u>	
	AL-11768-F		78	
	EA-96		6	
	AL-10747-A		6 vol% aromatic concentrate	
	Deionized Water		10	
<u>Fuel Temp,</u> <u>°C</u>	<u>Pressure on Secondary</u> <u>Fuel Filter, psig</u>		<u>Primary Fuel Filter</u> <u>Pressure Drop, in. Hg</u>	<u>Fuel Flow*</u>
	<u>Inlet</u>	<u>Outlet</u>		
-1	26	25	10	Sufficient
-2	38	38	11	Sufficient
-5	0	0	13	None

\* Fuel flow: ~125ml/min (sufficient to start a DD 6V-53T engine).

(SPEC22.A)

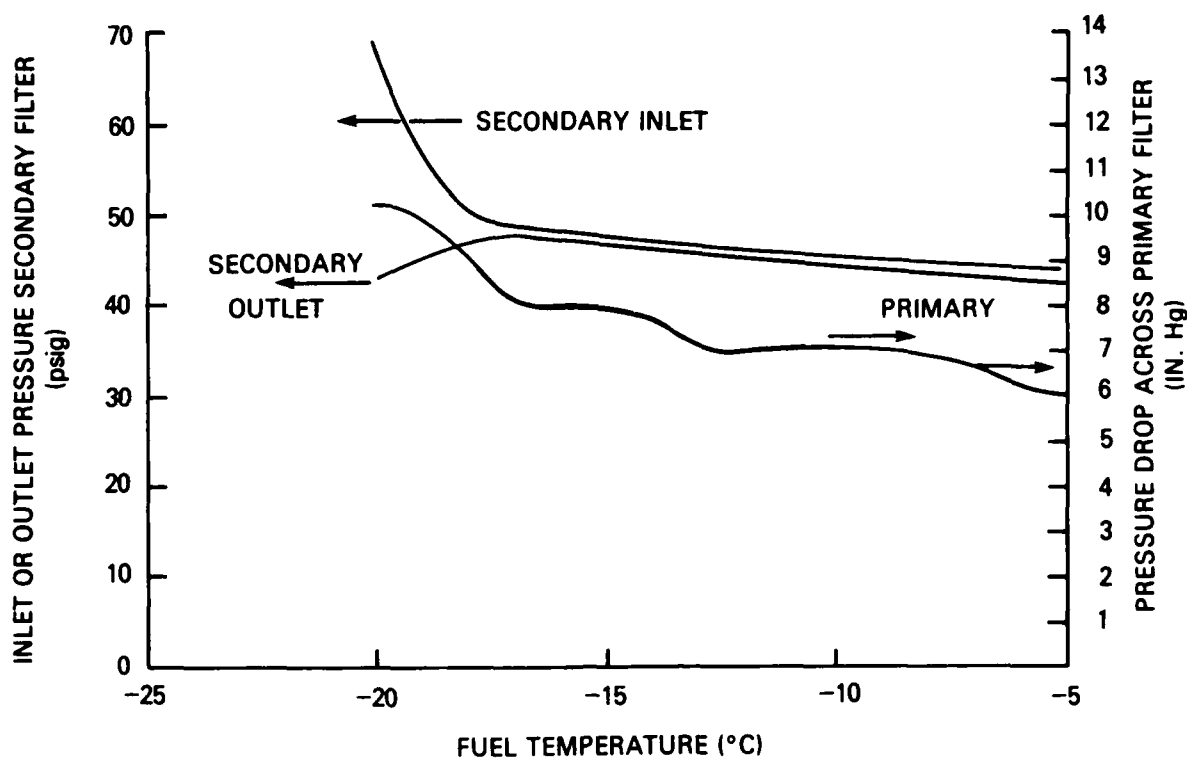


FIGURE 11. DIESEL FUEL-EMULSIFIER FRF MIXTURE WITHOUT WATER - LOW-TEMPERATURE FILTERABILITY IN SIMULATED DD 6V-53T FUEL SYSTEM

Table 22 presents the data on FRF blends of fuel, 7225, and of fuel, 11768, with and without aromatic concentrate, AL-10747. Examination of the data shows that, under this operating mode, all of these FRF blends had the filter-plugging temperatures of approximately  $-5^{\circ}\text{C}$ . Additional experiments were made with FRF-type blends, but the water content was reduced to 5 vol% and 2.5 vol% water. As shown in Table 23, the filter-plugging temperatures of 5 and 2.5 vol% water blends were approximately  $-5^{\circ}\text{C}$ , about the same as that of FRF containing 10 vol% water.

Follow-through experiments were conducted in which the filter housing on the suction side of the fuel pump was kept empty, and the sock-type filter was placed in the housing on the discharge side of the fuel pump. The fuel system was completely filled with FRF at room temperature and pump speed, and flows were adjusted to the idle conditions. The fuel pump was then turned off, and the temperature of the cold box was slowly lowered and allowed to equilibrate. Table 24 presents the data on pressures and flows

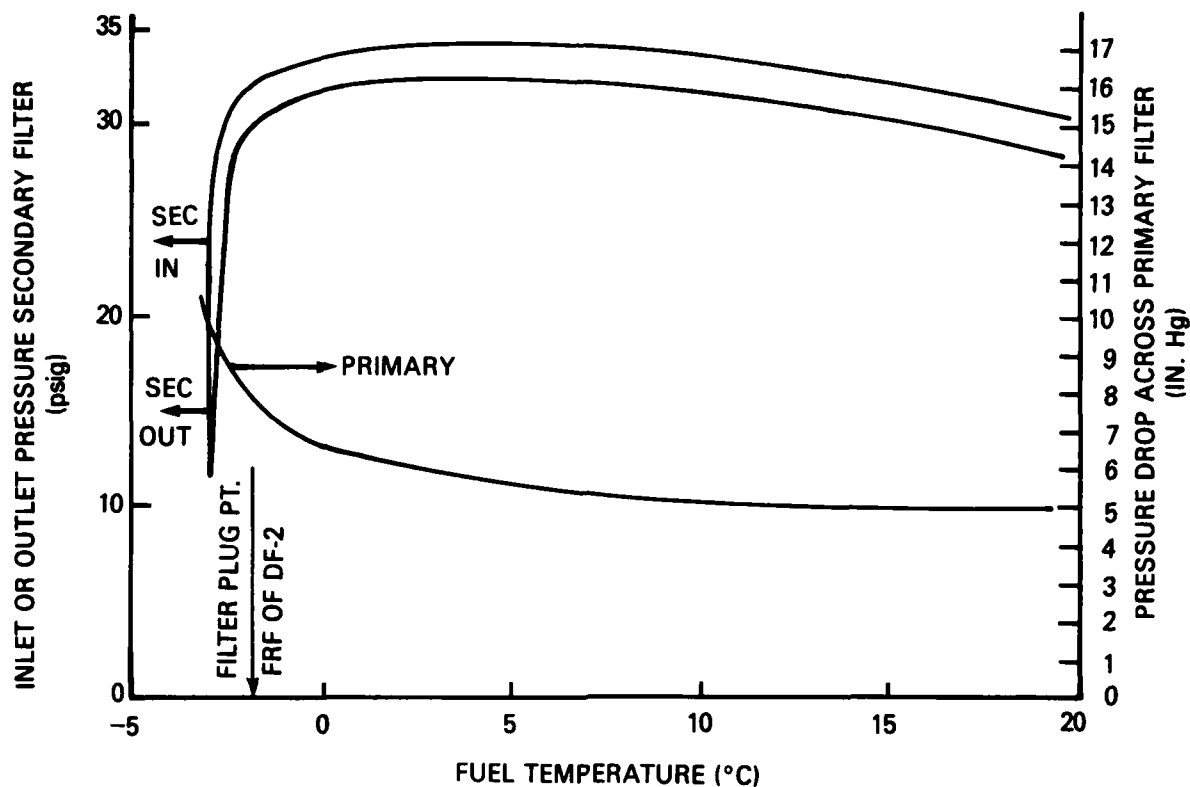


FIGURE 9. FRF OF DF-2 - LOW-TEMPERATURE FILTERABILITY IN SIMULATED DD 6V-53T FUEL SYSTEM

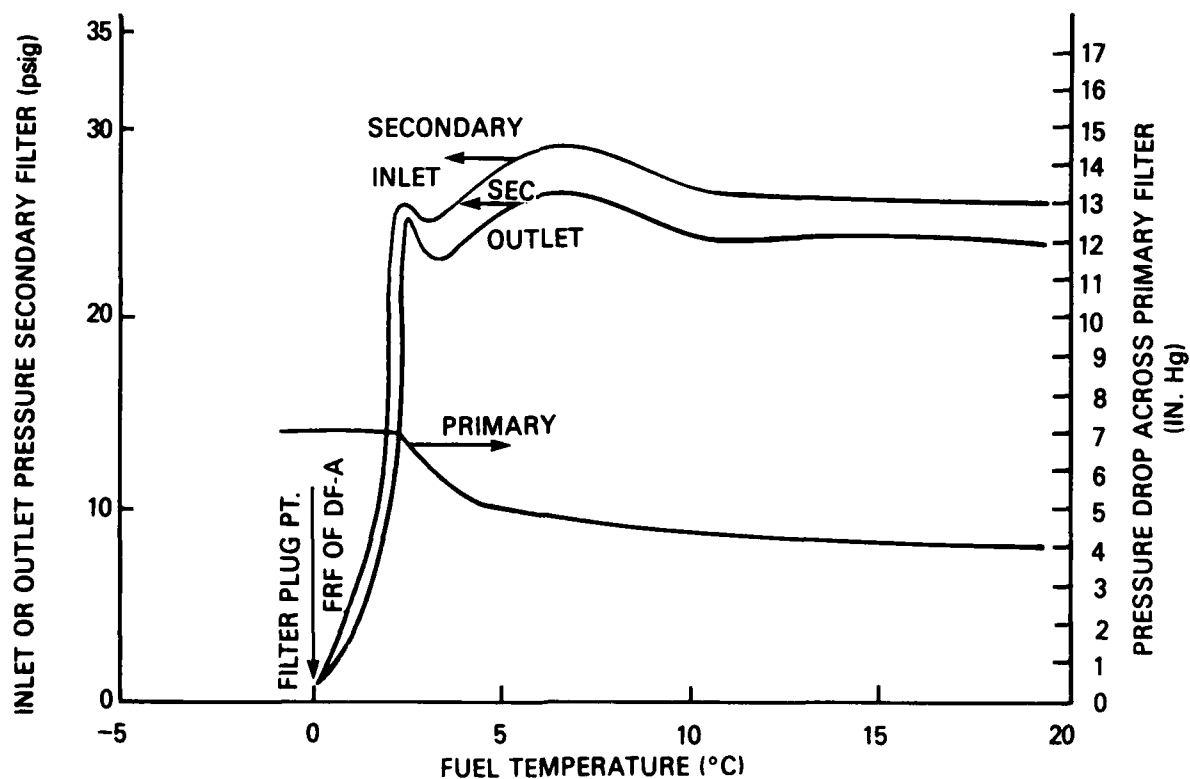


FIGURE 10. FRF OF DF-A - LOW-TEMPERATURE FILTERABILITY IN SIMULATED DD 6V-53T FUEL SYSTEM



Similar pressure-temperature curves for FRF compositions based on DF-A and DF-2 are shown in Figures 9 and 10. Both compositions experience limiting pumpability when the temperature is in the vicinity of the freezing point of water, due to primary filter plugging, as evidenced by very low inlet pressures to the secondary filter.

Following the observations of the foregoing tests, it was desirable to establish if the FRF surfactant alone could have caused the filter plugging observed with the FRF of DF-2 at  $-3^{\circ}\text{C}$ . In Figure 11, pressure drop temperature performance is shown for a DF-2/EA-99 mixture comprising 14:1 volume parts, respectively, the composition for FRF without water. The pressure drops are essentially the same as with base fuel, except that at the lower limit temperature (same as with base fuel,  $-20^{\circ}\text{C}$ ), the secondary filter plugging is responsible for reduced pumpability.

Minimum Temperatures for Maintaining Adequate Flow--Evaluations were conducted to realistically simulate pumpability in a fueled vehicle parked in a low-temperature environment with the engine off. The simulated DD 6V-53T engine fuel system was completely filled with the FRF at room temperature, and recycle and "consumption" flows were adjusted to idle conditions. The fuel pump was then turned off, and the temperature of the cold box was allowed to equilibrate at the desired temperature. If an attempt to start the system failed because a filter plugged immediately, the system was allowed to warm up to room temperature and the blend remixed in situ by recirculating it with the fuel pump. The system was again cooled to a somewhat higher equilibrium temperature with the pump off. If the attempt to start the system, with sufficient steady-state flow being delivered to the "injector side," was successful, the minimum temperature had been bracketed. To distinguish this experimental technique from the preceding pressure drop-temperature profiles, it is herein referred to as "equilibrium cooling."

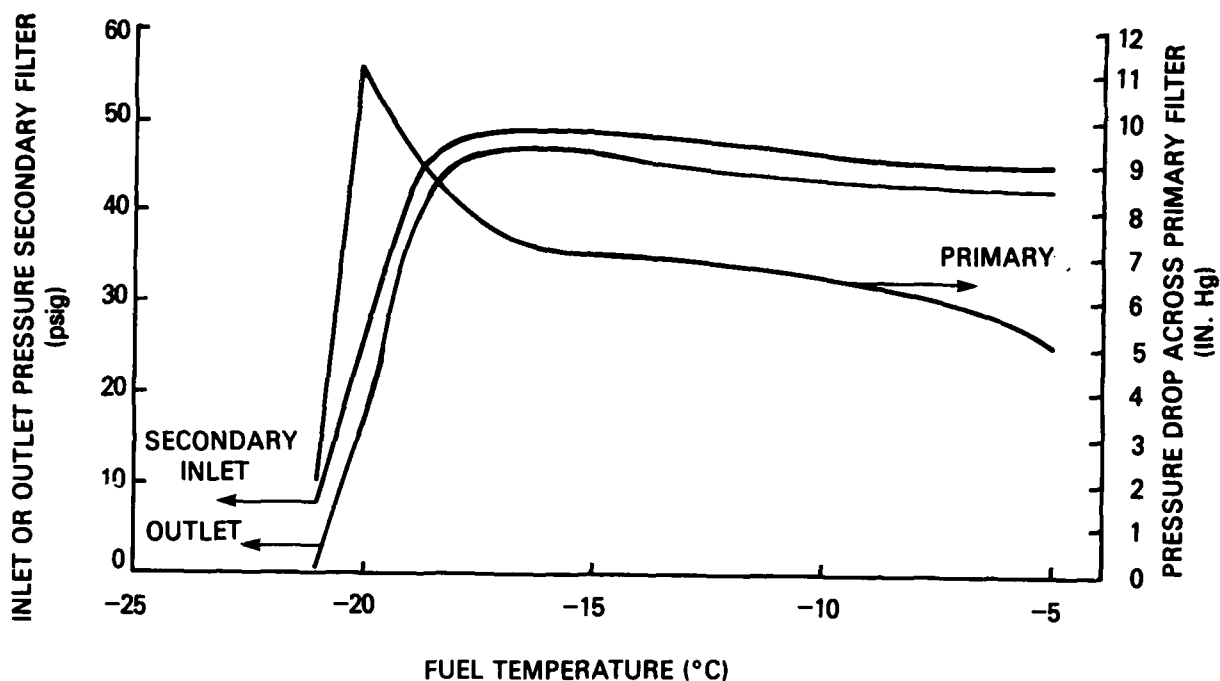


FIGURE 7. DF-2 BASE FUEL - LOW-TEMPERATURE FILTERABILITY IN  
SIMULATED DD 6V-53T FUEL SYSTEM

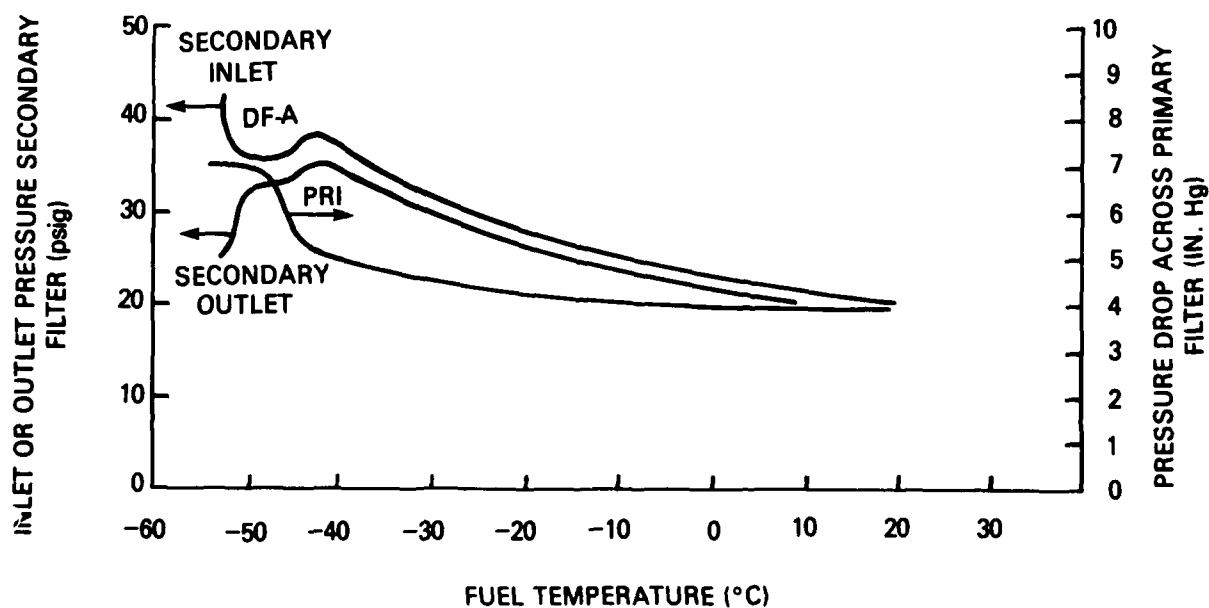


FIGURE 8. DF-A BASE FUEL - LOW-TEMPERATURE FILTERABILITY IN  
SIMULATED DD 6V-53T FUEL SYSTEM

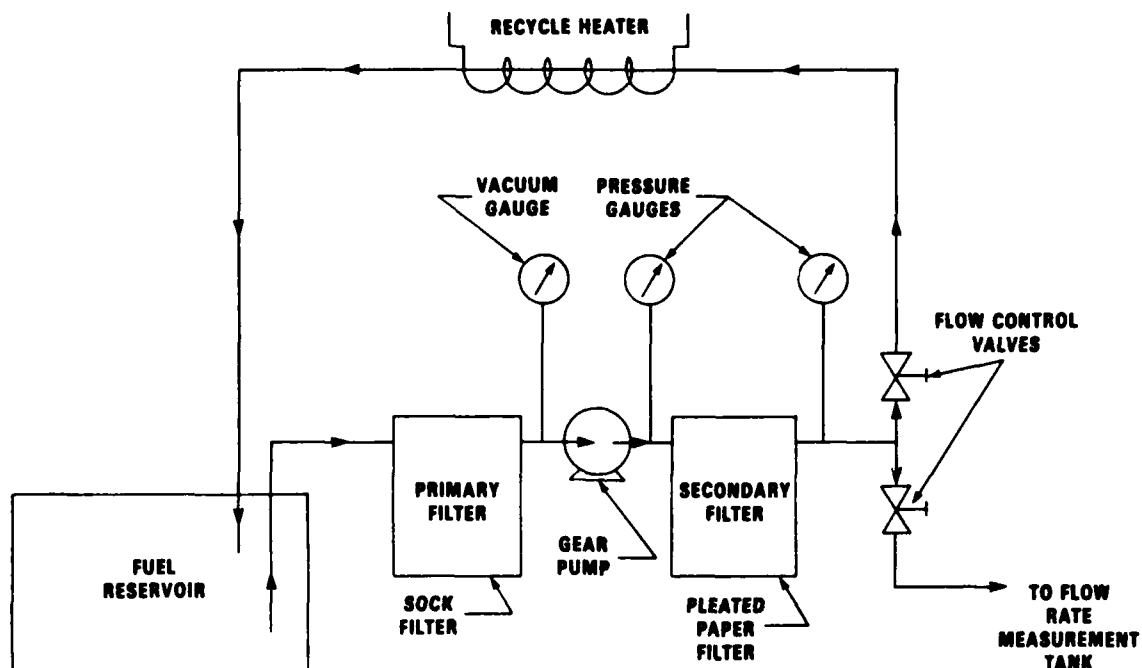


FIGURE 6. FLOW DIAGRAM OF CONTROLLED-TEMPERATURE DD 6V-53T  
ENGINE FUEL SYSTEM SIMULATOR

Figures 7 and 8 show pressure/temperature profiles across the primary and secondary filters for DF-2 (No. 7225) and DF-A (No. 9295) base fuels, respectively. Examination of Figure 5 shows that DF-2 plugs the primary filter at approximately  $-20^{\circ}\text{C}$ , essentially cutting the fuel from the secondary filter as evidenced by the very low inlet pressure on the secondary filter. DF-A fuel, however, is limited in its pumpability by the secondary filter at approximately  $-52^{\circ}\text{C}$ , as shown by very sharp differences in inlet and outlet pressures.

Similar pressure-temperature curves for FRF compositions based on DF-A and DF-2 are shown in Figures 9 and 10. Both compositions experience limiting pumpability when the temperature is in the vicinity of the freezing point of water, due to primary filter plugging, as evidenced by very low inlet pressures to the secondary filter.

TABLE 21. VISCOSITY OF FRF\*

<u>Temperature, °C</u>	<u>Maximum Reynolds Number</u>	<u>Viscosity, Centipoise</u>
20	580	4.66
15	380	5.82
10	310	6.55
5	220	7.65
0	150	9.13

\* Derived from flow through a smooth bore tubing.

through the FRF continues to flow well below 0°C. The viscosities derived from this type of measurement are more reproducible and reliable than those from the U-tube viscometers, and particularly so at the low temperatures.

#### Performance in Simulated Engine Fuel System:

Pressure Drop--Temperature Profiles During Continuous Cooling --Low-temperature FRF flow/filtration experiments were conducted in a controlled-temperature chamber using a continuous-flow, pumped loop. The loop was designed to serve as a simulator for a DD 6V-53T engine fuel system (4) and is illustrated in Figure 6. Prior to the availability of the "standard" base fuel (11768), two other base fuels and their FRF's were evaluated under the dynamic conditions of full recycle while temperatures dropped continuously. The system was filled at room temperature with 5 gallons of fuel, and idle speed was set to give 1650 ml/min flow totally recycled to the fuel tank. The chamber air, and thus the fuel tank, temperature was then allowed to drop continuously until the filter plugged. Typically, the rate of cooling averaged about 10°C per hour. It should be noted that the low-temperature box did not have a programmable controller to change the rate of cooling; however, the operator could select and change the set point of the controller so that there was less than 2°C difference between the air temperature and the fuel temperature during the cooling.

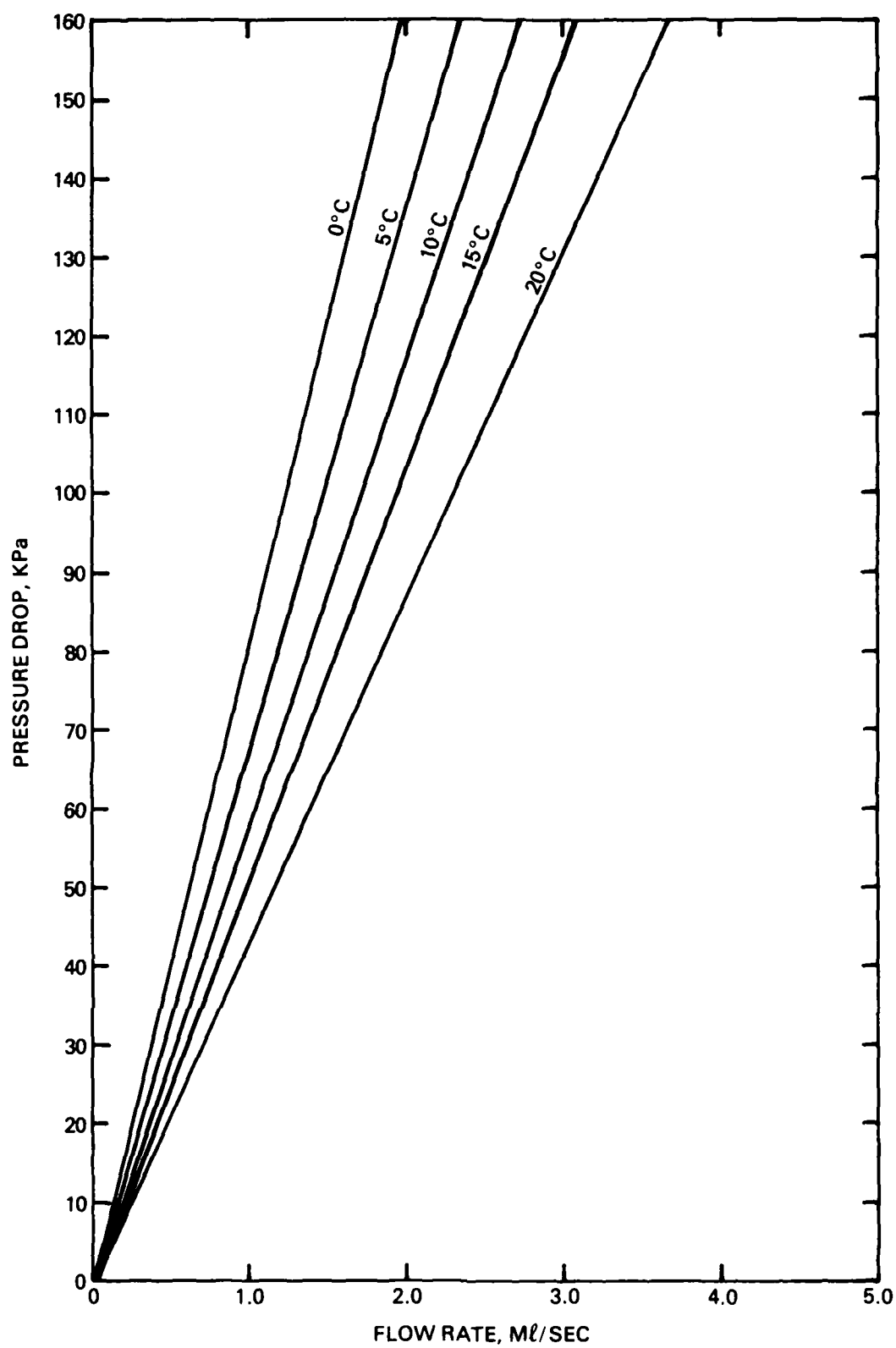


FIGURE 5. FLOW PROPERTIES OF FRF

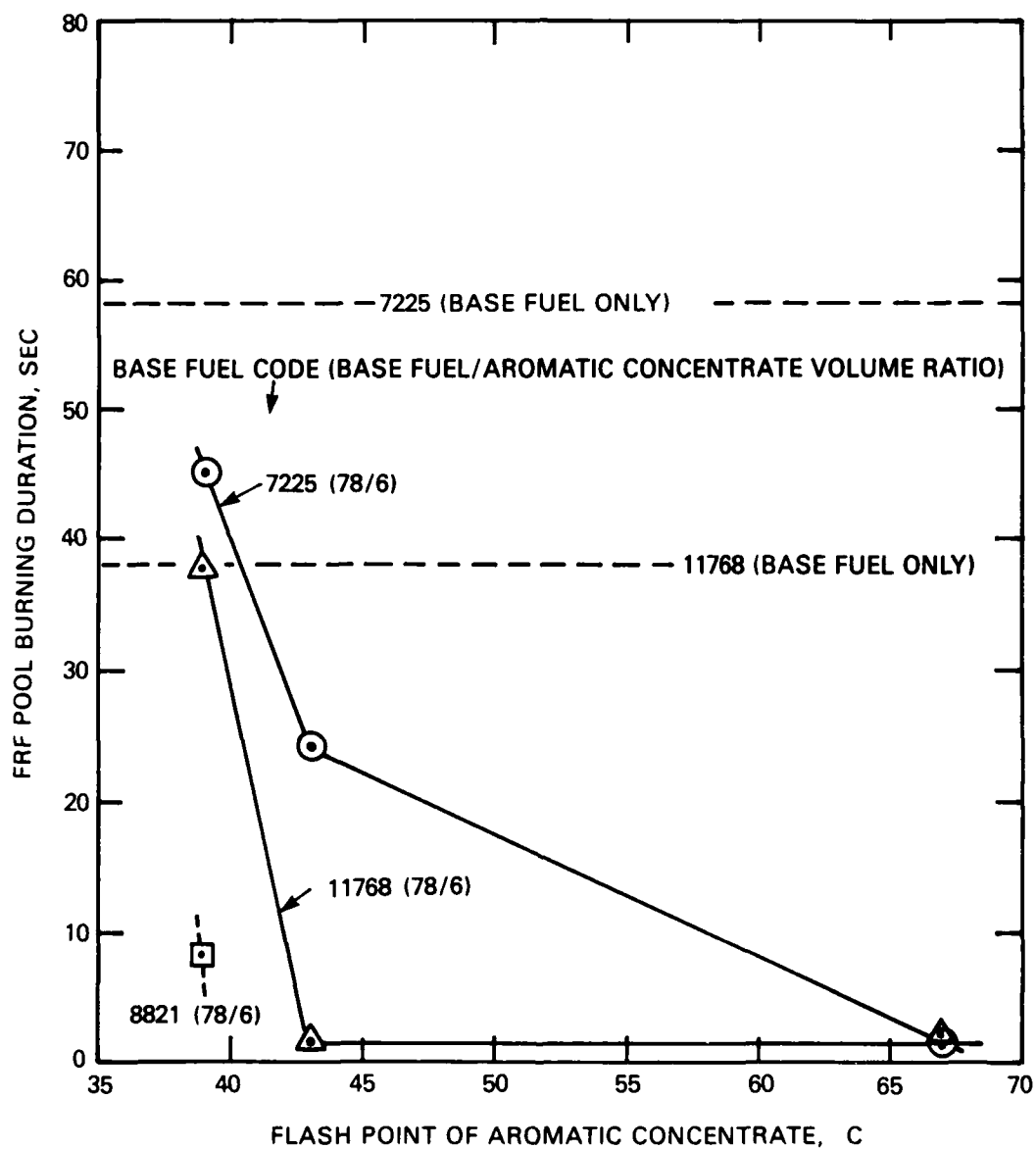


FIGURE 4. INFLUENCE OF FLASH POINT OF AROMATIC CONCENTRATE  
ON FRF SELF-EXTINGUISHMENT TIME

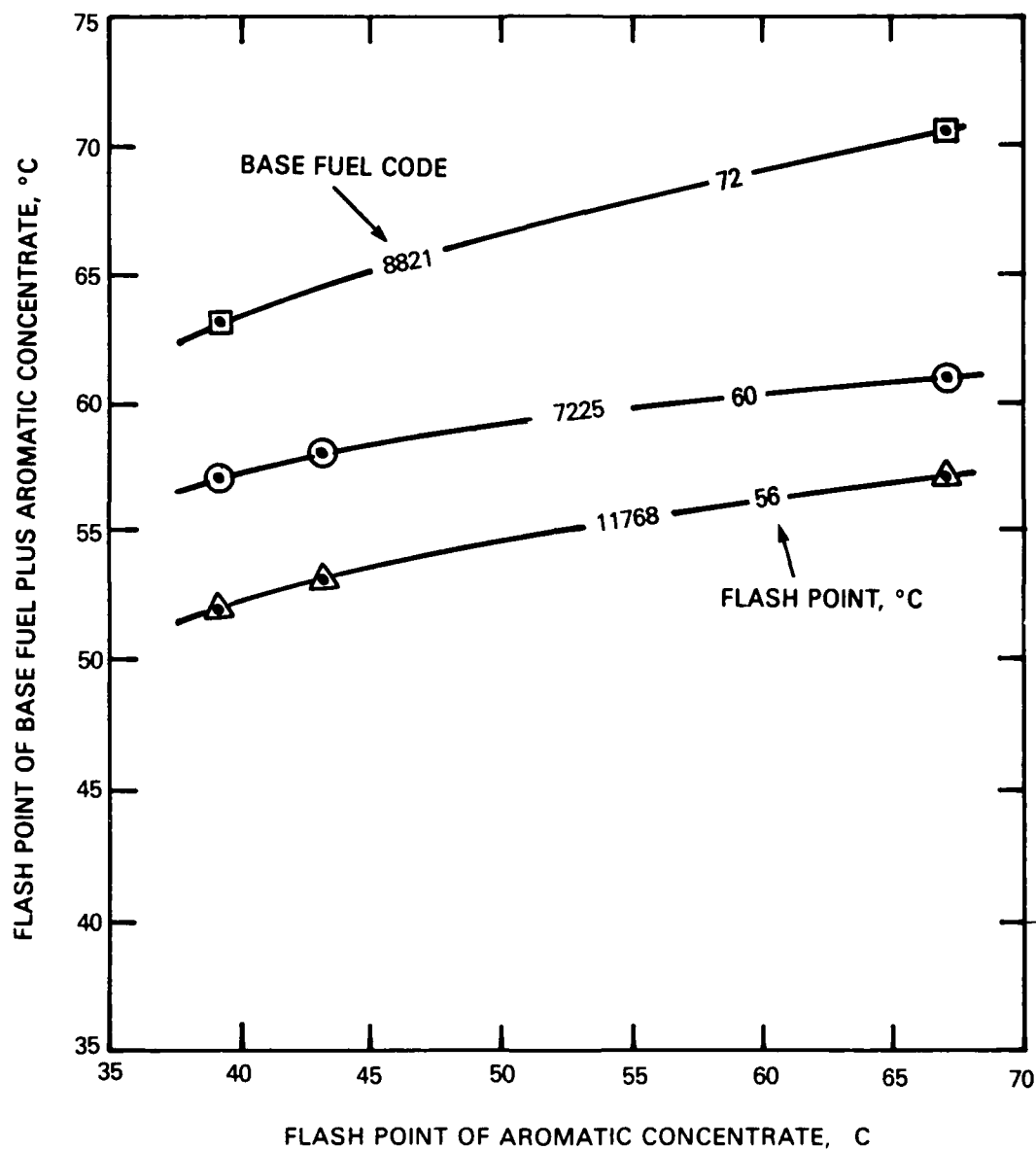


FIGURE 3. INFLUENCE OF FLASH POINT OF AROMATIC CONCENTRATE ON FLASH POINT OF BASE FUEL/AROMATIC CONCENTRATE MIXTURE

TABLE 20. SUMMARY OF FLASH POINT AND IMPACT DISPERSION TEST RESULTS  
FOR THREE BASE FUELS AND THREE AROMATIC CONCENTRATES

7225	Base Fuel		Vol Parts				FA 96	Tap Water**	Flash Point*, °C	Time to Self- Extinguishment, Sec
	8821	11768	Aromatic Concentrate		10716	10748	10749			
100	--	--	--	--	--	--	--	--	60	58 (avg of 2)
--	100	--	--	--	--	--	--	--	72	--
--	--	100	--	--	--	--	--	--	56	38
--	--	--	100	--	--	--	--	--	39	--
--	--	--	--	100	--	--	--	--	67	--
--	--	--	--	--	100	--	--	--	43	--
78	--	--	6	--	--	--	--	--	57	--
78	--	--	--	6	--	--	--	--	61	--
78	--	--	--	--	--	6	--	--	58	--
78	--	--	--	6	--	--	6	--	62	--
78	--	--	--	--	--	--	6	--	59	--
--	78	--	6	--	--	--	--	--	63	--
--	78	--	--	6	--	--	--	--	71	--
--	--	78	6	--	--	--	--	--	52	--
--	--	78	--	6	--	--	--	--	57	--
--	--	78	--	--	--	6	--	--	53	--
--	--	78	--	6	--	--	6	--	58	--
--	--	78	--	--	--	--	6	--	56	--
78	--	--	6	--	--	--	6	10	--	45
--	78	--	6	--	--	--	6	10	--	8
--	--	78	6	--	--	--	6	10	57	38
78	--	--	--	6	--	--	--	10	--	1.5
78	--	--	--	--	--	--	6	10	--	24
--	--	78	--	--	--	6	--	10	--	1.9
--	--	78	--	--	--	--	6	10	--	1.3
84	--	--	--	--	--	--	6	10	--	1.2 (avg of 6)

\* Pensky-Martens Closed Cup Flash Point; ASTM D 93

\*\* ~ 300 ppm TDS



TABLE 26. ADDITIVES TRIED FOR IMPROVING  
LOW-TEMPERATURE PROPERTIES

FRF Composition (Vol%)	Emulsion Ratings, Time After Preparation			
	(Room Temperature)			At 0°C
	20 min	2 hr	20 hr	2 hr
AL-11768-F (79) + EA-96 (6) D.I. H <sub>2</sub> O (10) Sec. Butanol (5)	3	6	6	6
AL-11768-F (79) + EA-99 (6) D.I. H <sub>2</sub> O (10) Sec. Butanol (5)	3	6	--	--
AL-11768-F (79) + EA-96 (6) D.I. H <sub>2</sub> O (10) Tert. Butanol (5)	2	3	6	6
AL-11768-F (79) + EA-99 (6) D.I. H <sub>2</sub> O (10) Tert. Butanol (5)	3	6	--	--
AL-11768-F (79) + EA-96 (6) D.I. H <sub>2</sub> O (10) n-Amyl Alcohol (5)	3	6	--	--
AL-11768-F (79) + EA-99 (6) D.I. H <sub>2</sub> O (10) n-Amyl Alcohol (5)	3	6	--	--

minute, the run was considered "no start." The temperature was then raised slightly and the test repeated until a start was obtained.

The results obtained with this series of experiments provided some indication, not only of low-temperature ignition quality of FRF, but also an assessment of the fuel-handling system response to FRF blends at low temperatures. This information is extremely important since viscosity measurements made on these blends at 0°C and 10°C indicated that the fuel becomes very viscous.

Table 27 lists the cetane numbers and minimum starting temperatures for the various blends. Addition of the surfactant alone decreased the cetane number by 5 numbers but did not impair startability. When the 10 vol% water was added, the cetane number decreased by 8 more numbers and, as expected, the minimum starting temperature increased somewhat. Addition of 0.25 percent cetane improver restored about 3 numbers; 0.5 percent, 5 numbers (normal maximum additive); 0.75 percent, 7 numbers; and 1.0 percent, 9 numbers. These results indicate that the loss in cetane number exhibited by FRF relative to that of the base fuel can be restored by addition of a typical cetane improver. However, unexpectedly, the cetane number impaired performance by further increasing the minimum starting temperature. Incidentally, the fuel flow rate for the FRF blends at these lower temperatures was similar to that of the base fuel, apparently indicating that the fuel system, which did not contain a filter, was not being plugged.

Full-Scale Vehicle Tests--Arrangements were made by Belvoir R&D Center for FRF full-scale vehicle evaluations to be conducted by Yuma Proving Ground personnel. These tests were made with an M113A1 armored personnel carrier and an M60A3 battle tank. The fuel that was tested had been shipped from Tank Automotive Command (TACOM) and was from the same batch that had been procured for the AVDS 1790 engine endurance test of FRF.(4) The FRF blend had been prepared at TACOM with the prototype continuous blending system (which has since been shipped back to this laboratory).

TABLE 27. EFFECT OF CETANE IMPROVER\* ON CETANE NUMBER AND STARTING TEMPERATURE IN AN INSULATED DD 4-53T ENGINE

Fuel Sample	Cetane Number	Starting Temperature, °C**
Base Fuel (10200)	54.8	-9 to -8
Base Fuel + 6% Surfactant (EA-99)	49.6	-10
FRF (of 10200 base fuel)	41.3	-6
FRF + 0.25% Cetane Improver	44.0	-2
FRF + 0.50% Cetane Improver	46.0	-2
FRF + 0.75% Cetane Improver	48.3	-2
FRF + 1.0% Cetane Improver	50.1	-5

\* Cetane improver, DII-3 (2-ethylhexylnitrate)

\*\* Starting temperatures represent the average of two no-start airbox temperatures and two start temperatures that are no more than 2°C apart.  
ND = Not determined.

Analyses of the base fuel and the FRF that was prepared from it are summarized in Appendix B (Table B-2). It may be noted that the base fuel contained high concentrations of aromatic hydrocarbons and that it failed the accelerated stability specifications. The produced FRF microemulsion (made with Detroit tap water (180 ppm total dissolved solids)) contained the recommended 10 vol% water concentration, but only 5.3 vol% of emulsifying agent (EA-96) instead of the recommended 6 vol%. Loss in gravimetric net heat of combustion was found to be 11.9 percent (9.7 percent on volumetric basis) and cetane number loss was 7 units. Measured kinematic viscosity of the FRF was within VV-F-800 specifications at 40°C, and was still acceptable at 20°C. However, at 10°C, the measurements gave erratic results that averaged 54±8 cSt. It has been shown previously (1) that the U-tube capillary viscometer is not suitable for FRF at low temperatures. This is an artifact of the experimental method.

The vehicular evaluations that were performed are described as follows:

1. Maximum speed tests were conducted over an essentially flat test course that was approximately 2 miles long. There was a slope of approximately 1

degree in the track; therefore, all tests were repeated in both directions. The surface of the test course appeared similar in appearance to an aggregate surface highway.

2. Accelerations from 0-20 mph were conducted over the same test track and were also repeated in both directions.

3. Drawbar horsepower pull tests were conducted on the same test track, and horsepower pull was measured using a wheeled vehicular dynamometer built by FMC.

Tables 28 and 29 list the results that were obtained.

TABLE 28. COMPARISON OF MAXIMUM VEHICLE SPEEDS

Vehicle	Maximum Speed (mph)			% Loss
	Base Fuel	FRF	Loss	
M113A1	39.0	34.6	4.4	11
M60A3	31.4	30.7	0.7	2

TABLE 29. COMPARISON OF VEHICLE ACCELERATION RATES

Vehicle	Time to Accelerate From 0 to 20 mph (sec)			
	Base Fuel	FRF	Loss	% Loss
M113A1	10.0	13.2	3.2	32
M60A3	14.7	17.5	2.8	19

The drawbar horsepower measurements were taken over a range of road speeds and, generally, show an average of 8 to 10 percent power loss with the M113A1 and an average of 4 to 6 percent with the M60A3. It was interesting to note that the drawbar pull tests showed the M60A3 to be more efficient at higher speeds (2 percent loss) which appears to concur with the loss in maximum speed of only 2 percent.

### Special Observations and Comments--

1. The amount of smoke produced by the two fuels was noted since laboratory engines had indicated a reduction in smoke. It was observed that a definite reduction of smoke occurred with the FRF under the test conditions.

2. From a practical standpoint, loss of maximum speed may not be a penalty in actual use for the following reason: Field tests (going from point A to point B) have demonstrated that maximum speeds cannot be used very often since the type of terrain which the off-road vehicles travel over drastically limits the speeds at which a vehicle can be safely handled. Therefore, maximum speeds can seldom be used in actual field conditions.

### Lubricity Properties:

Lubricity tests were conducted on a ball-on-cylinder machine (BOCM) fabricated according to drawings furnished by Exxon Research and Engineering. The test apparatus consists of a fixed 1/2-inch diameter ball held in a chuck and forced vertically downward against the highest point on the surface of a 1-3/4 inch diameter rotating cylinder. A more complete description and discussion are presented in Reference 7.

Criteria defining satisfactory or unsatisfactory fuel lubricity as measured by the BOCM are generally unavailable; however, the U.S. Navy has established tentative guidelines for JP-5 fuel. These are shown in Table 30, along with experimental data for base fuel and its FRF. Based on these results, no serious wear problems are indicated with the use of FRF.

TABLE 30. FUEL LUBRICITY

<u>Fuel</u>	<u>Wear Scar Diameter, mm</u>
Good*	≤0.42
Marginal*	0.43-0.48
Poor*	>0.49
FRF Base Fuel (AL-10200)	0.26
FRF	0.38

\* U.S. Navy definitions

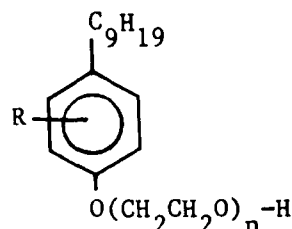
### III. BASIC RESEARCH ON FIRE-RESISTANT DIESEL FUEL- EMULSIFIER MODIFICATION

This program was initiated to develop information on the effects of emulsifier chemistry on aqueous diesel fuel emulsion properties such as phase stability, low-temperature behavior, and tolerance toward both varied fuel composition and dissolved electrolytes in the water. This study was undertaken to identify those aqueous water-in-oil (W/O) type microemulsion systems that would alleviate the problems associated with the current FRF composition while preserving the desirable fire resistance characteristics. Several approaches were taken to achieve these goals, including an extensive literature survey(8).

The experimental work included several approaches that are summarized in this section.

During the first phase of this investigation, the principles of Wade (9) were followed. According to his approach, pairs of surfactants are used and scans are made to find optimum salinity at which maximum volume of "middle phase" is formed. These middle phases are said to be true microemulsions that are in equilibrium with a water rich lower phase and a hydrocarbon rich upper phase. The surfactant blend that produces the largest middle phase is the optimal surfactant composition for the particular hydrocarbon (mixture) and the electrolyte solution. It was reported that increasing the optimal surfactant blend to water ratio will yield a homogeneous, stable microemulsion.

In this work, pairs of the commercially available ethoxylated nonylphenols and dinonylphenols of various hydrophilic chain lengths were investigated (Igepal CO and DM series by GAF, respectively). These compounds may be described by the following formulas:



where

R = H for the CO series;

R =  $\text{C}_9\text{H}_{19}$  for the DM series;

n = number of moles of ethylene oxide per mole of alkylphenol.

Depending upon n, the number of moles of ethylene oxide per mole of alkylphenol, the hydrophile--lipophile balance (HLB) of the product may be adjusted. Initially, the HLB region between 9 and 15 were investigated.

Several series of experiments were performed. In each series, diesel fuel (No. 8821) was used as the hydrocarbon. Scans were made using single surfactants and blends of surfactants, where the water's electrolyte (NaCl) concentration was varied between 0 and 10,000 ppm. The phase behavior of the resultant mixtures were examined at 30, 40, and 50°C. There were no conditions found under which any of the listed products yielded usable emulsions.

Further, it was noted by Rosen and Li (10) that there is a dynamic equilibrium among the three phases discussed by Wade, in which no single phase may exit without the other two. This finding also implied that increased surfactant-to-water ratio will not yield a homogeneous emulsion, as required by the FRF concept. These same researchers (10) investigated the composition of a middle phase emulsion made with an anionic surfactant, phenoxyethanol as cosurfactant, heptane, and aqueous sodium chloride solution. The composition was studied as a function of distance from the upper and lower interfaces. They found that upon standing, a surfactant-cosurfactant gradient and a density gradient developed, indicating that the middle-phase emulsions are nonhomogeneous.

For these reasons, the Wade approach was abandoned.

Emulsification of aqueous electrolyte (NaCl) solutions in a hydrocarbon (diesel fuel No. 8821) in the presence of one of the following four emulsifying agents was also attempted:

- (a) sodium di-(2-ethylhexyl) sulfosuccinate (Aerosol-OT)
- (b) oleic acid diethanolamide with excess diethanolamine (EA-99)
- (c) lauric acid diethanolamide, with excess diethanolamine
- (d) capric acid diethanolamide, with excess diethanolamine

Each of the last three products is a so-called "Kritchevsky amide," as they were synthesized by reacting one mole of the acid with two moles of diethanolamine. By this reaction, formation of side products is suppressed, with the products containing only a mixture of the amide, soap, and the amine. (In emulsification, the amine is postulated to serve as a cosurfactant). While EA-99 has been the "standard" emulsifying agent in the preparation of FRF, the lauric and capric acid derivatives could have beneficial effects, as they are more hydrophilic products than EA-99.

In each of the emulsification experiments, the electrolyte (NaCl) was used at various concentrations between 0 and 10,000 ppm; blends were made at hydrocarbon-to-aqueous phase volume ratios of 2:1, 1:1, and 1:2. The resultant mixtures were equilibrated at 30°, 40°, and 50°C. Under these baseline experimental conditions studied, i.e., using only a "single" emulsifying agent, EA-99 is superior.

The effects of using combinations of two emulsifying agents were studied. The basis of this study is the widely accepted practical fact that a combination of surfactants may produce emulsions of superior quality. The phenomenon may be explained the following way. For producing an emulsion from two mutually insoluble liquids, a "coupling," i.e., an emulsifying agent is necessary. This emulsifying agent must have good solubility in each of these liquids. While such single surfactants may exist for specific systems, normally it is easier to find a pair (or mixture) of surfactants to

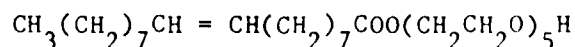


emulsify the mutually insoluble liquids. Coupling of these phases is made possible by the fact that each surfactant is soluble in one of the liquids, and the two surfactants are soluble in each other.

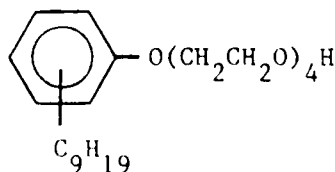
Pairs of Kritchevsky amides were used to emulsify aqueous sodium chloride solutions in a mixture of hydrocarbons, namely, diesel fuel No. 8821. Surfactant mixtures were made at 10 vol% intervals of oleic acid diethanolamide (EA-99) and either capric acid diethanolamide or lauric acid diethanolamide. Sodium chloride concentration in water was varied between 0 and 500 ppm. The emulsified phases were allowed to reach equilibrium at 30° and 40°C. Visual appearance of the various blends indicates possible improvement over using EA-99 alone. Specifically, while EA-99 seems to be the preferred surfactant at salinity values of 0 and 100 ppm, a 4:1 mixture of EA-99 to capramide gives improved phase behavior at sodium chloride concentrations of 250 and 500 ppm at temperatures of 30°C and 40°C.

Encouraged by the results obtained using the binary blends of surfactants, additional combinations of surfactants were also explored. These included ternary blends of surfactants whose efficacy in emulsification was studied. The effects of four surfactants (from GAF) were studied:

- a) A polyethoxylated oleic acid, containing five oxyethylene groups (Emulphor VN-430)



- b) A polyethoxylated nonylphenol, containing four oxyethylene groups (Igepal CO-430)



- [illegible]

- It was shown that, when used alone, none of the listed surfactants produced a water-in-oil emulsion. Two ternary mixtures of surfactants were made, each containing an equivolume quantity of products. Both blends contained the polyethoxylated oleic acid and the anionic phosphate ester. Additionally, one of the blends also contained the dinonyl derivative, while the other blend contained the monononyl compound. The studied salinity range in the water was between 0 and 500 ppm NaCl. Surfactant mixture to electrolyte volume ratios ( $\alpha$ ) were set at 1:1 and 1:2. Emulsion stabilities were observed between 0° and 40°C. Both ternary surfactant blends gave transparent microemulsions between 0° and 50°C at  $\alpha = 1$ . Increasing the electrolyte concentration to give  $\alpha = 0.5$  showed that, under the studied experimental conditions, the dinonyl derivative (DM-430) is a better emulsifying agent than the monononyl (CO-430) derivative, as the latter gave 0.2 to 0.9 vol% phase separation at 40°C, while the use of the dinonyl derivative yielded transparent emulsions under all the studied conditions in fuel No. 8821.

(SPEC22.A)

To extend the useful temperature range of the microemulsions to below 0°C, modifications of the solubility of the surfactant blend was investigated. To avoid separation of water (ice) from the emulsions, surfactants with increased water solubility was attempted. As nonionic surfactants usually exhibit increased solubility at lower temperatures, while anionic surfactants behave "normally," an increase in the relative proportion of the anionic part of the ternary blend was done. From the original 1:1:1 volume ratios for the three surfactants, the ratio of the anionic phosphate ester was increased to 1.25, 1.5, 1.75, 2.0, 5.0, and 10.0. The total surfactant blend to water volume ratio was held at 1.0. Sodium chloride content of the water was varied between 0 and 500 ppm. Experimental results indicated that increasing the anionic surfactant's relative volume ratio to above 2 prevented microemulsion formation, but in no case did it improve low-temperature behavior.

To reduce the complexity of this ternary surfactant blend, attempts were made to incorporate the chemical structural features of the three surfactants into one compound. This preliminary work did not yield acceptable results.

It was concluded that the studied ternary surfactant blend produced an aqueous microemulsion from diesel fuel No. 8821 that exhibited a lower kinematic viscosity at 0°C than the product made by the use of the Kritchevsky amide of oleic acid and diethanolamine. However, even with this surfactant system, the useful low-temperature limit was above -5°C, while the upper temperature limit was reduced from over 40°C to approximately 30°C.

In another attempt to lower the temperature sensitivity and reduce the low-temperature viscosity of aqueous fuel emulsions, the effects of various alcohols were also studied.

The necessity of the inclusion of an alcohol cosurfactant in microemulsions has been questioned.(11,12). However, the majority of workers in this field (13,14) have used various low-molecular weight alcohols to stabilize aqueous microemulsions. (It is believed that the diethanolamine component of the FRF emulsifier acts as a cosurfactant in that system).

In this study, the effects of various paraffinic alcohols on the phase stability and the temperature dependence of kinematic viscosity of aqueous diesel fuel emulsions were studied. The studied systems comprised a diesel fuel (No. 8821), a surfactant (EA-99, the Kritchevsky amide of oleic acid and diethanolamine), water, and the alcohol as a cosurfactant. The order of addition of the ingredients was surfactant - alcohol - water - diesel fuel. The alcohol solubilized the surfactant in water so the order was not important. In each case, an equivolume (5 vol% each) of surfactant and water was used, and the concentration of the alcohol was varied between 0 and 5 vol%. The studied alcohols included iso-propanol, tert-butanol, cyclohexanol, 1-pentanol, hexanol, 1-heptanol, 1-octanol, decanol, and 1,4-cyclohexanediol. The experimental results are tabuled in Table 31. The following conclusions were drawn from these studies:

- There is a maximum amount of alcohol that is tolerated by the system. The exact amount depends upon the specific alcohol used.
- Reversible phase separations were observed by lowering the sample temperature.
- Formation of ice crystals was observed as the emulsion's temperature was lowered to  $-5^{\circ}\text{C}$ .
- Kinematic viscosity values given in this table are average values for up to 14 determinations. In each case, in which the standard deviation was found to be over 0.05, the consequent determinations gave progressively lower values. As yet, there is no explanation for this phenomenon.
- While addition of iso-propanol and tert-butanol increased the kinematic viscosity values of the emulsions, the addition of cyclohexanol decreased these values. This fact is considered significant, as such relatively low viscosity values have not been achieved before this time.
- Addition of 0.5 vol% of any of the alcohols from pentanol to decanol lowered the kinematic viscosity relative to that of the alcohol-free emulsion at the measurement temperatures of  $0^{\circ}$ ,  $10^{\circ}$ , and  $20^{\circ}\text{C}$ .

Compositional behavior was studied at  $10^{\circ}$  and  $30^{\circ}\text{C}$  of the system comprising diesel fuel (No. 8821), surfactant (EA-99), and water containing 0, 100, and

TABLE 31. EFFECT OF ALCOHOLS ON THE KINEMATIC VISCOSITY OF W/O EMULSIONS

Alcohol Name	Vol%	Vol%		Rating*	Kinematic Viscosity, cSt, at °C			Notes
		8821	Defolized Water		20	10	0	
---	---	100	---	N/A	5.09	6.84	10.20	
1-Propanol	0.5	90.0	5	1	9.14	17.97	54.56	
1-Propanol	1.0	89.5	5	1	12.18	63.11	137.42	
1-Propanol	1.5	88.5	5	1	21.14	Separated	---	
1-Propanol	2.0	88.0	5	6	---	---	---	Reversible Phase Separation
1-Propanol	2.5	87.5	5	6	---	---	---	Lower Phase = 39 vol%
1-Propanol	5.0	85.0	5	6	---	---	---	Lower Phase = 31 vol%
1-Butanol	0.5	89.5	5	1	9.52	42.91	186.45	Lower Phase = 26 vol%
1-Butanol	1.0	89.0	5	1	12.38	40.98	88.06	Lower Phase = 19 vol%
1-Butanol	1.5	88.5	5	1	12.85	34.15	Separated	
1-Butanol	2.0	88.0	5	1	13.21	Separated	---	Reversible Phase Separation
1-Butanol	2.5	87.5	5	2	13.28	Separated	---	Reversible Phase Separation
1-Butanol	5.0	85.0	5	6	---	---	---	Lower Phase = 29 vol%
c-Hexanol	0.5	89.5	5	1	7.70	11.59	24.00	
c-Hexanol	1.0	89.0	5	1	7.68	10.39	16.14	
c-Hexanol	1.5	88.5	5	1	7.53	10.74	16.07	
c-Hexanol	2.0	88.0	5	5	---	---	---	
c-Hexanol	2.5	87.5	5	5	---	---	---	
c-Hexanol	5.0	85.0	5	6	---	---	---	Lower Phase = 3 vol%
1-Pentanol	0.5	89.5	5	1	7.08	9.70	14.30	
1-Pentanol	1.0	89.0	5	1	7.16	9.62	13.75	
1-Pentanol	1.5	88.5	5	6	---	---	---	
Hexanol	0.5	89.5	5	1	7.13	9.47	13.72	
Hexanol	1.0	89.0	5	5	---	---	---	
Hexanol	1.5	88.5	5	6	---	---	---	
1-Heptanol	0.5	89.5	5	1	7.08	9.51	13.73	
1-Heptanol	1.0	89.0	5	6	---	---	---	
1-Octanol	0.5	89.5	5	1	7.12	9.59	13.61	
1-Octanol	1.0	89.0	5	5	---	---	---	
Decanol	0.5	89.5	5	1T	7.03	9.43	13.79	
Decanol	1.0	89.0	5	5	---	---	---	
1,4-c-Hexanediol	0.5	89.5	5	1	25	47	125	
1,4-c-Hexanediol	1.0	89.0	5	6	---	---	---	

Notes: \*Rating of visual appearance of the emulsion at room temperature

1. Transparent microemulsion
2. Translucent microemulsion
3. Whitish-brown macroemulsion
4. Whitish-yellow macroemulsion
5. Emulsion with trace of cream ( $\leq 0.5$  vol%)
6. Emulsion with less than 2 vol% cream

6. Phase separation

500 ppm of sodium chloride. The graphically illustrated results, obtained 24 hours after the preparation of the blends, are reproduced in Figures 12 through 17, where the apexes represent 100% of oil (o), water (w), and surfactant (s), respectively. In Figures 12-14, the data observed at 30°C are summarized for sodium chloride concentrations of 0, 100, and 500 ppm in water, respectively. The corresponding data for 10°C are given in Figures 15-17. On each diagram, the compositions that gave transparent microemulsions are shaded. Other results of visual observation are also shown on the diagrams, such as the presence of hazy or translucent microemulsions, macroemulsions of milky appearance, phase separation, and gel formation. Liquid crystal formation, as indicated by birefringence (different refractive index in different directions), was also noted.

Examination of the diagrams indicate that--with the specific components used--at 30°C essentially identical regions of transparent microemulsions exist. These regions are independent of electrolyte concentration within 0 and 500 ppm. Observations at 10°C indicate an expanding envelope of transparent microemulsion region as the electrolyte concentration increased from 30°C. In each case described here, a straight line may be drawn that connects the 100 percent oil ("O") apex and the 85:10:5 = oil:water:surfactant (volume ratio) compositional data point. This line describes a boundary below which transparent microemulsions do not exist. It may be noted that the last generation of fire-resistant diesel fuel (FRF) may be described by a point on these diagrams that is just above this line, inside the shaded envelope.

It remains unresolved how the compositional behavior of such systems would change if the observations were made after the presently used 24-hour time period, and how they depend upon storage temperatures, higher electrolyte concentrations, different electrolytes, and--most importantly--upon the chemical composition of the diesel fuel itself.

#### Conclusions:

These exploratory studies of variations of emulsifier chemistry did not yield microemulsion systems which promise alleviation of the problem areas

A discussion is given of fuel blending costs and anticipated R & D costs. It is mentioned that, in addition to the estimated hardware savings, a reduction in battlefield casualties will also result, and that the supply lines (storage tanks, tank trucks, pipe lines, etc.) will be less vulnerable to fire, thus increasing survivability.

A requirement is anticipated for five additional personnel per petroleum supply company, or a total of 250 more personnel for 50 blending units, Army wide.

System Development includes five separate plans.

The operational support plan involves conducting studies of concepts and operational testing with MOS and troop units using fuel consuming equipment.

The technical development plan includes additional testing to be conducted and coordinated by USAMERADCOM and the Propulsion Systems Laboratory to address critical issues not yet answered by research. The testing will include investigation of the effects of fire-resistant fuel on engine starting over the same temperature range as that encountered by diesel fuel; effects on fuel economy, maximum power, and speed; effects on engine fuel systems; effects on reliability, availability, maintainability, and durability of engines and fuel systems; and effects on smoke generation equipment, crew heaters, and petroleum handling equipment. New quality assurance test methods required for fire-resistant fuel will be identified, and additional petroleum laboratory testing equipment requirements will also be identified. The fire-resistant fuel filterability, handling, and storage characteristics will be established, and the effects of centrifugal pumps on such fuels will be investigated. Any introduced toxic hazard to personnel will be identified. Trade-off analyses will determine the most cost/operational effective combination of fire-resistant fuel characteristics and engine/equipment changes required. Changes in RAM of vehicle systems are to be established. Because of its passive nature, fire-resistant fuel falls in the "RAM-not-required" category; however, the listed critical issues are to be resolved before or during testing (DT-I).

The logistic support plan will consider minimum servicing requirements, maximum ease of maintenance, and the use of standard components. Logistics Support Analysis/Record (LSA/LSAR) data will be used to identify specific logistic requirements. Performance and maintenance data, potential logistic problem areas, and logistic support parameters will be estimated based on present systems while considering known technological improvements and other factors. The logistic support plan must be available for evaluation at DT/OT-I.

A training support concept will be required as with any other similar system. The support plan must be available for evaluation at OT-I.

The personnel support plan requires that the material developer project personnel and training requirements, pay particular attention to simplicity, human factors, and reliability of automatic features. The DARCOM critical task analysis will be utilized to determine information needed to assess

LETTER OF AGREEMENT  
FOR  
FIRE-RESISTANT FUEL AND EQUIPMENT SYSTEMS  
FOR COMBAT VEHICLES (ACN 51140)  
27 May 1980

Executive Summary

The Army's need for a fire-resistant diesel fuel and equipment system, for ground combat vehicles is discussed. It is noted that such a fuel will reduce the vulnerability of combat vehicles to fire, increase crew survivability, reduce the number of combat vehicles lost due to fuel fires, reduce repair cost and time, and increase the number of repairable vehicles.

The operational concept involves the shipment of diesel fuel into the theater by ocean tanker to be offloaded into onshore storage facilities. The fuel will be treated with additives to render it fire resistant. The equipment required to treat the diesel fuel will be operated by a petroleum supply company as far to the rear as practicable and no further forward than the corps class III supply point. Both untreated diesel fuel and fire-resistant diesel fuel will be usable by all diesel fuel burning equipment within the division without a significant reduction in power, performance, RAM, or fuel economy of fuel-consuming systems.

The system description includes definition of the requirements for a blending unit as follows: It must be skid mounted, have 300 gallons per minute capacity, and be capable of being transported by C-130, C-141, C-5A, commercial cargo ship, and 2-1/2 ton or larger truck. All components within the unit are to be commercial items with collective RAM characteristics that are acceptable, and couplings are to be compatible with current fuel handling equipment. The system is to be capable of being used, stored, and transported in hot, basic, cold, and severe cold climate types. The treated fuel to be produced by the blending system is described as: being capable of mixing with untreated diesel fuel in a vehicle fuel system without adverse effects; not degrading the performance of current military diesel or turbine engines below their design specifications; and having a low-temperature operating limit roughly the same as the cloud point of normal diesel fuel. Requirements for health and personnel hazard standards are specified, and coordination efforts are defined to assure international interoperability and standardization.

Itemization of Prospective Operational Effectiveness and Cost includes a discussion of extensive studies reported in "Combat Operations Loss and Expenditure Data-Vietnam" (COLED-V). These data revealed that about 8 percent of the armored personnel carriers (APC) destroyed could have been saved from total destruction with the use of an effective fire prevention system. Such vehicles experiencing a sustained fuel fire were beyond rebuild at any level of maintenance, leading to the assumption that such a vehicle not gutted by fire could be repaired.





APPENDIX A

EXECUTIVE SUMMARY OF LETTER OF AGREEMENT FOR FIRE-RESISTANT  
FUEL AND EQUIPMENT SYSTEMS FOR COMBAT VEHICLES

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7. Russell, J.A.; Cuellar, J.P.; Tyler, J.C.; et al., "Development of Accelerated Fuel-Engines Qualification Procedures Methodology, Vol. 1," U.S. Army Fuels and Lubricants Research Laboratory, U.S. Army Contract No. DAAK70-81-C-0209, Report AFLRL No. 144, December 1981 (Government Accession No. A113461).

Tests will be conducted to demonstrate the efficacy of fuel filter heating for alleviating the low-temperature filter plugging problems. Such tests will employ low-temperature engine fuel system simulators and cold room vehicular tests. Full-scale FRF mixing tests will be conducted, and these will include low-temperature environments. The performance of an AGT-1500 turbine engine with FRF will be evaluated to assess the impact of FRF.

TABLE 32. FRF USER REQUIREMENTS--RATING SUMMARY

Requirements	FRF Pass/Fail Rating
• Crew survivability resulting from use of FRF must be significantly improved.	(+)*
• Equipment survivability/repairability cost effectiveness resulting from use of FRF must be improved.	(+)*
• FRF must be usable worldwide under all weather conditions.	(-)
• FRF must not increase costs significantly.	(-)
• FRF must be compatible with present fuel handling and distribution systems.	(-)
• FRF must be compatible with elastomers in fuel handling and distribution system.	(±)*
• FRF must not need a dedicated distribution system.	(-)
• FRF must not require fuel handling and distribution system modification.	(-)
• FRF must not require vehicle fuel system modification.	(+)
• FRF must be compatible with normal vehicle fuel system, fuel handling and distribution system contaminants.	(+)
• FRF must not result in more than minimal additional fuel handling and distribution system equipment/personnel requirements.	(-)
• FRF must not cause metal corrosion.	(+)
• FRF must not degrade engine power/range below design specifications.	(±)
• FRF must not cause filter plugging at subzero (°C) ambient temperatures down to the cloud point of the base fuel.	(-)
• FRF must not require water purification.	(-)
• FRF must have satisfactory storage stability for at least six months.	(+)
• FRF emulsifier premix and water must produce satisfactory microemulsions with any specification DF-A, DF-1, DF-2, or NATO F-54 diesel fuel.	(+)
• FRF must remain stable when diluted with other specification diesel fuels.	(+)
• FRF must be compatible with automatic fire detection/suppression systems.	(?)
• FRF must be suitable for non-engine diesel fuel consuming equipment.	(+)*

Legend: (+) meets requirement; (-) does not meet requirement; (±) marginal (yes and no); (?) unknown; (\*) requires confirmation.

(WDW04,E)

encountered with the current FRF. In fact, the collective results of this experimental study and the parallel literature review indicate that the Kritchevsky amide originally selected for FRF may represent optimum emulsifier chemistry for yielding stable aqueous diesel fuel microemulsions.

#### IV. STATUS OF FRF DEVELOPMENT

All of the critical issues listed in Table 1 (p. 11) have not yet been satisfactorily resolved. FRF does not satisfy all of the requirements specified by the military users. These requirements were established during the on-going FRF research by: the listings in the LOA; additional requisites surfacing during special meetings among the military developer and military FRF users; and requirements generated by other sources. Table 32 itemizes comparisons of these user-stipulated requirements versus the present status of FRF pass/fail ratings.

The most significant FRF inadequacy is the filter plugging it causes at subfreezing temperatures. This, in turn, precludes FRF use worldwide under all weather conditions. The need for substantial additional equipment and personnel is determined predominantly by the deployment concept specified in the LOA. Reduction of the required storage life and/or water content could possibly decrease water purity requirements. Furthermore, use of FRF only in vehicles anticipating combat service could substantially reduce total quantities of water and surfactant needed. Collectively, such reductions in the quantity of water and surfactant and the purity of water, if achievable, could substantially reduce the costs and the need for additional equipment and personnel.

Work is ongoing to address the above described problems associated with using the current FRF formulation, and to those critical issues which have not been adequately assessed. In particular, the following areas will be addressed.

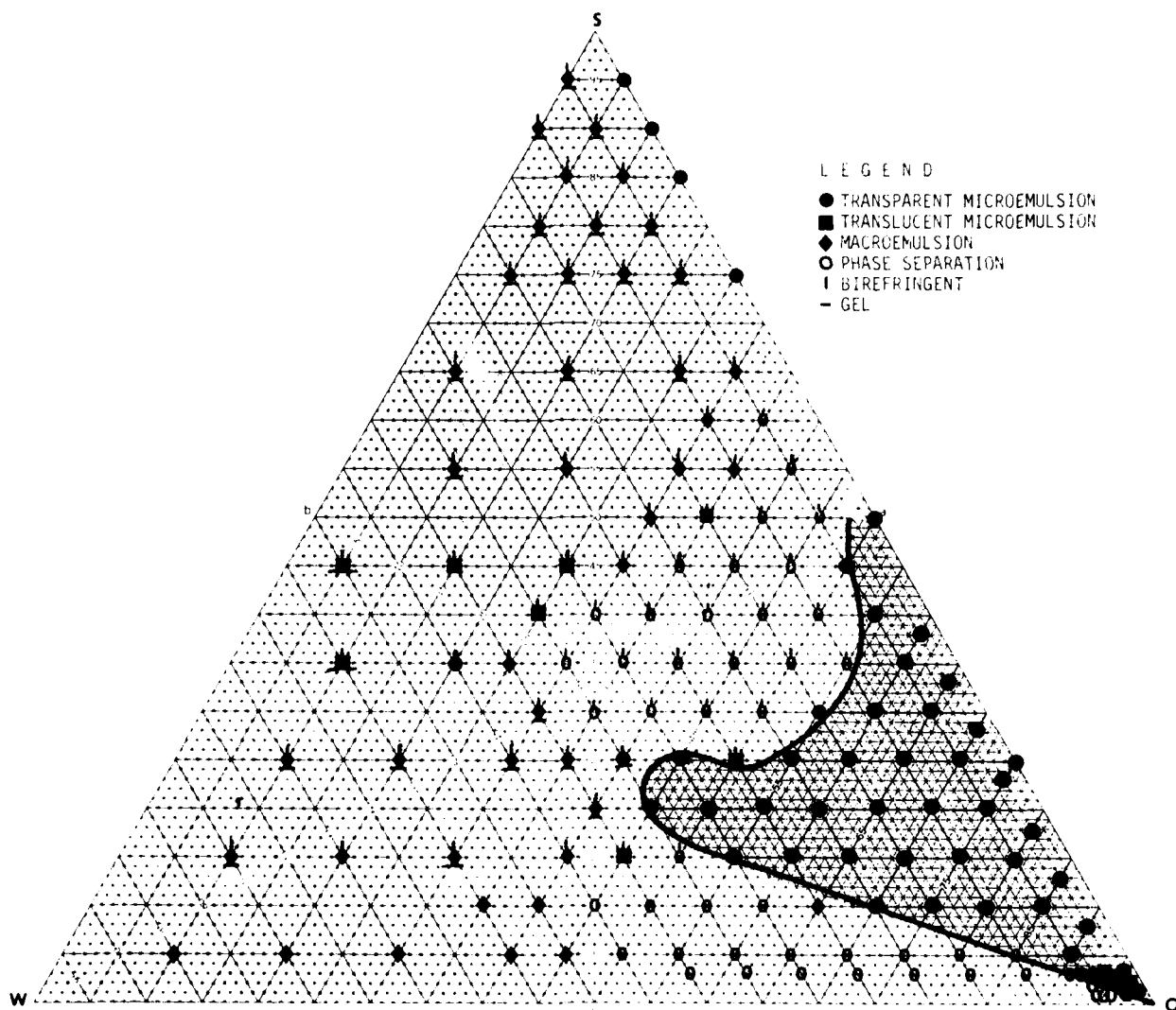


FIGURE 17. COMPOSITIONAL BEHAVIOR DIAGRAM OF DIESEL  
FUEL/EA-99/500 PPM NaCl SOLUTIONS at 10°C

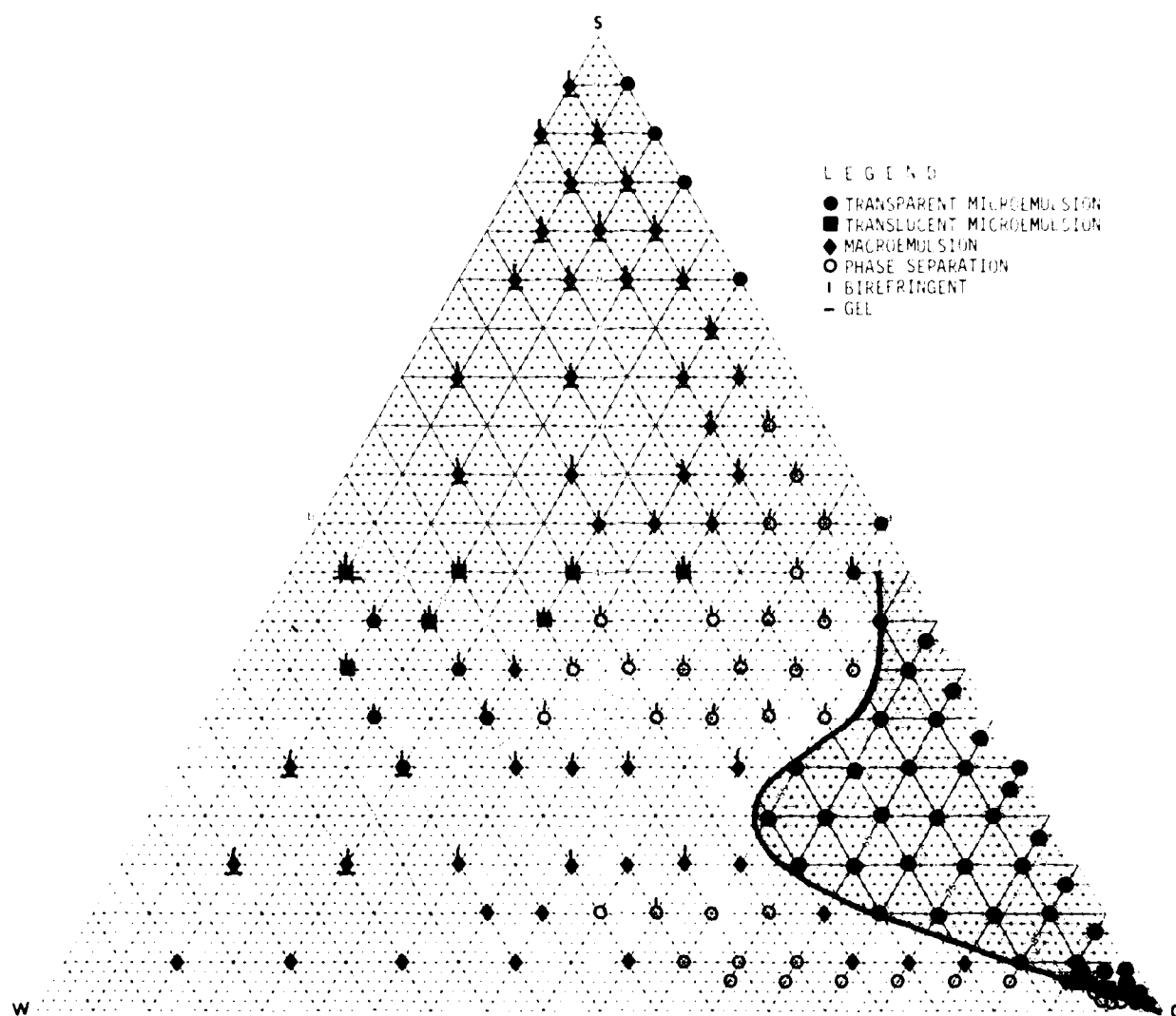


FIGURE 16. COMPOSITIONAL BEHAVIOR DIAGRAM OF DIESEL FUEL/EA-99/100 PPM NaCl SOLUTIONS AT 10°C



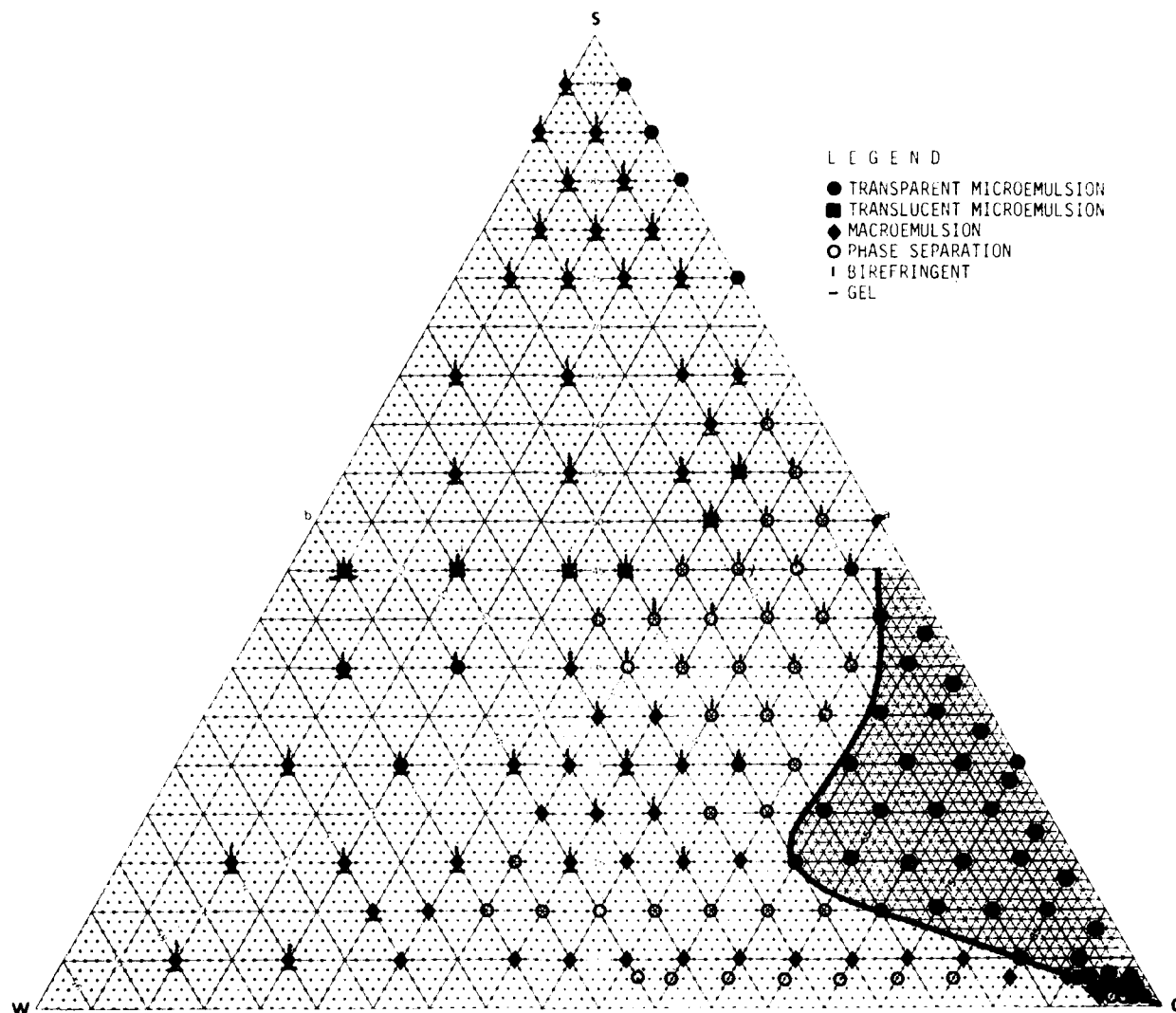


FIGURE 15. COMPOSITIONAL BEHAVIOR DIAGRAM OF DIESEL  
FUEL/EA-99/0 PPM NaCl SOLUTIONS AT 10°C

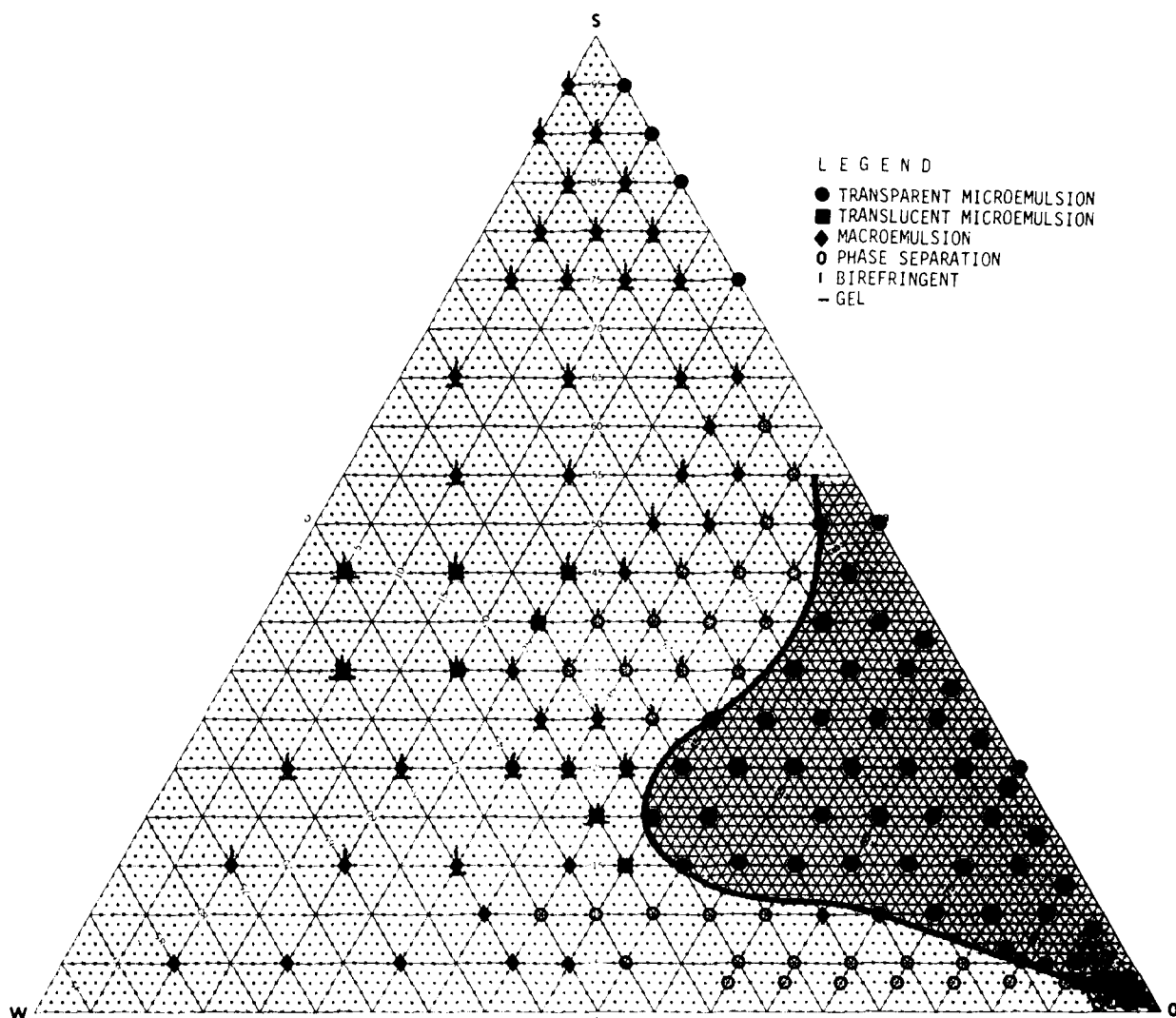


FIGURE 14. COMPOSITIONAL BEHAVIOR DIAGRAM OF DIESEL FUEL/EA-99/500 PPM NaCl SOLUTIONS AT 30°C

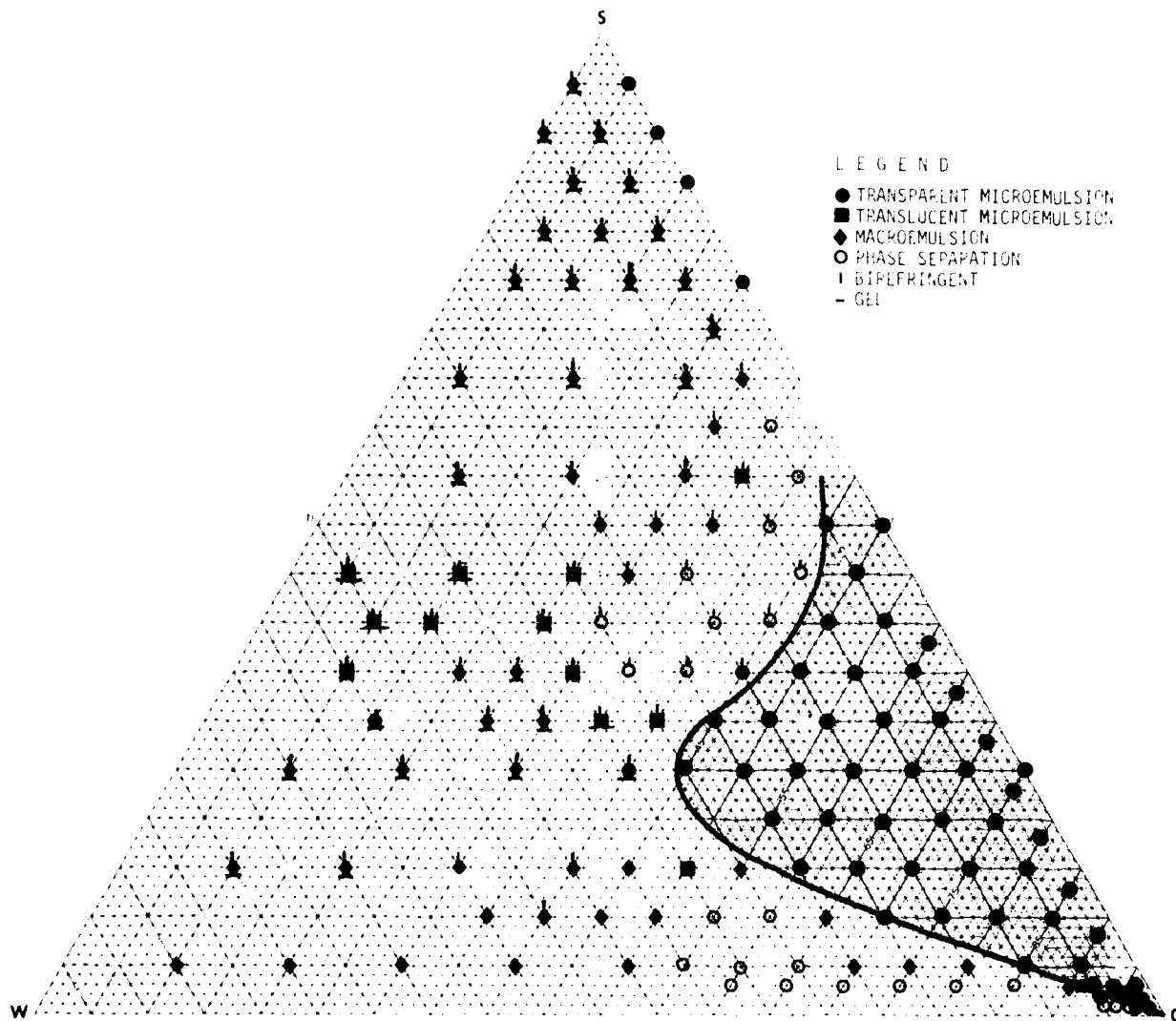


FIGURE 13. COMPOSITIONAL BEHAVIOR DIAGRAM OF DIESEL  
FUEL/EA-99/100 PPM NaCl SOLUTIONS AT 30°C

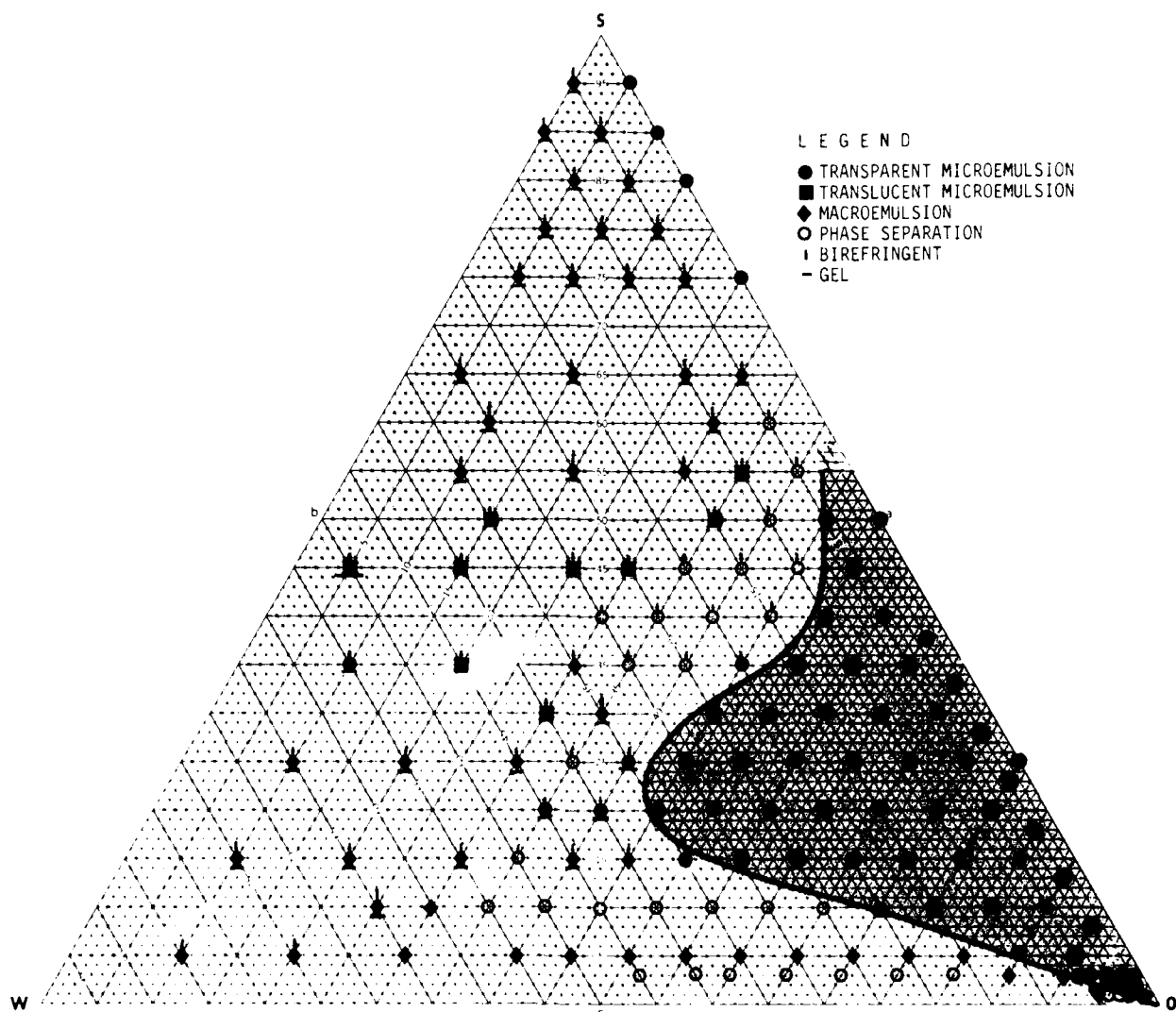


FIGURE 12. COMPOSITIONAL BEHAVIOR DIAGRAM OF DIESEL FUEL/EA-99/0 PPM NaCl SOLUTIONS AT 30°C

organizational and personnel implications of including the system in the total force structure. The personnel support plan must be available for initial evaluation at DT/OT-I.

The Schedule and Milestones lists the LOA approval as 2QFY80; test equipment purchase as 2QFY81; test equipment DT/OT-I as 2QFY82; and VAL IPR as 1QFY83.

The Funding is summarized in a tabular cost assessment section of the LOA.

APPENDIX B  
PROPERTIES OF FUELS USED IN THIS STUDY

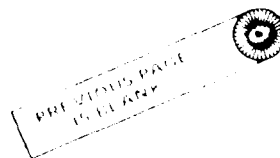


TABLE B-1. PROPERTIES OF FUELS USED IN THIS STUDY

Property	Test Method	DF-1 (9294)	DF-A (9295)	DF-2 (7225)	DF-2 (8821)	DF-2 (110700)	DF-2 (11798)	DF-2 (8512)	DF-2 (12443)	DF-2 (12443)	DF-2 (12443)
Gravity, °API at 15.5°C	287	42.2	47.7	36.1	35.2	37.2	37.2	36.2	37.2	37.2	37.2
Density, g/ml at 15.5°C		0.8146	0.7879	0.8438	0.8484	0.8404	0.8404	0.8404	0.8404	0.8404	0.8404
Flash point, °C	93	82	45	60	77	49	49	77	77	77	77
Cloud point, °C	2500	-23	-52	-21	-1	-	-	-	-	-	-
Pour point, °C	97	-35	-56	-24	-10	-	-	-	-	-	-
Color	1500	--	--	--	--	--	--	--	--	--	--
Kinematic viscosity, cSt, at 20°C	445	--	--	--	--	--	--	--	--	--	--
Kinematic viscosity, cSt, at 40°C	445	1.93	1.18	2.20	3.20	--	--	3.20	3.20	3.20	3.20
Steam jet num, mg/100 ml	381	--	--	3.9	3.2	--	--	3.2	3.2	3.2	3.2
Accelerated stability, mg/100 ml	2274	--	--	0.6	2.7	--	--	2.7	2.7	2.7	2.7
Copper strip corrosion	130	1A	1A	1A	1A	--	--	1A	1A	1A	1A
Total acid number, mg KOH/g	664	--	--	0.01	0.03	--	--	0.03	0.03	0.03	0.03
Heat of combustion, gross, Btu/lb	240	19955	19810	19427	19670	--	--	19587	19587	19587	19587
Heat of combustion, gross, MJ/kg	240	46.42	46.08	45.1	45.7	--	--	45.56	45.56	45.56	45.56
Heat of combustion, net, Btu/lb	240	--	--	18283	18450	--	--	18450	18450	18450	18450
Heat of combustion, net, MJ/kg	240	--	--	42.5	42.8	--	--	42.6	42.6	42.6	42.6
Aromatics by HPLC, wt%	--	--	--	27.5	25.9	--	--	--	--	--	--
Aromatic ring carbon, wt%, by UV	--	--	--	--	--	--	--	--	--	--	--
monocyclic	--	3.4	5.5	7.1	7.5	7.6	7.6	7.5	7.5	7.5	7.5
dicyclic	--	6.7	2.4	11.5	6.5	4.8	4.8	6.5	6.5	6.5	6.5
tricyclic	--	0.09	0.0	0.3	0.4	0.2	0.2	0.4	0.4	0.4	0.4
total	--	10.2	7.9	18.9	14.4	12.6	12.6	14.4	14.4	14.4	14.4
Water, wt%, by Karl Fischer	1744	--	--	--	--	--	--	--	--	--	--
Sulfur, wt%, by XRF	--	0.02	<0.01	0.35	0.47	--	--	0.47	0.47	0.47	0.47
Carbon, wt%	3178	--	--	86.8	86.7	--	--	86.7	86.7	86.7	86.7
Hydrogen, wt%	3178	--	--	13.2	13.3	--	--	13.3	13.3	13.3	13.3
Cetane no.	613	60	51	48	51	--	--	51	51	51	51
Distillation, °C	86	--	--	--	--	--	--	--	--	--	--
IBP	--	212	164	166	183	--	--	183	183	183	183
5% evap.	--	217	175	--	--	--	--	--	--	--	--
10% evap.	--	219	178	218	225	--	--	225	225	225	225
20% evap.	--	221	181	--	--	--	--	--	--	--	--
30% evap.	--	223	182	--	--	--	--	--	--	--	--
40% evap.	--	226	185	--	--	--	--	--	--	--	--
50% evap.	--	229	188	244	282	--	--	282	282	282	282
60% evap.	--	233	191	--	--	--	--	--	--	--	--
70% evap.	--	240	195	--	--	--	--	--	--	--	--
80% evap.	--	252	199	--	--	--	--	--	--	--	--
90% evap.	--	278	205	294	231	--	--	231	231	231	231
95% evap.	--	303	223	--	--	--	--	--	--	--	--
EP	--	325	252	358	361	--	--	361	361	361	361
Recovered, %	--	99.0	99.0	--	--	--	--	--	--	--	--
Residue, %	--	1.0	1.0	--	--	--	--	--	--	--	--
Loss	--	0.0	0.0	--	--	--	--	--	--	--	--

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TABLE B-2. ANALYSIS OF VV-F-800B-DF-2 DIESEL FUEL (AFLRL NO. 10135)  
AND THE FRD MADE FROM IT AT TACOM

Property	Test Method ASTM D	Specification Value	Base Fuel	FRF*
Gravity, °API at 15.5°C	287	--	34.2	29.7
Density, g/mL at 15.5°C	--	--	0.8535	0.8773
Flash point, PMCC, °C	93	--	61	None**
Fire point, COC, °C	92	--	95	--
Cloud point, °C	2500	--	-16	--
Pour point, °C	97	--	-36	--
Kinematic viscosity, cSt, at 40°C	445	2.0-4.3***	2.39	4.07
Kinematic viscosity, cSt, at 20°C	445	--	--	9.3
Kinematic viscosity, cSt, at 10°C	445	--	--	54
Kinematic viscosity, cSt, at 0°C	445	--	6.38	--
Accelerated Stability, mg/100 mL	2274	1.5	2.86	--
Steam jet gum, mg/100 mL	381	--	6.7	--
Copper strip corrosion	130	3	1A	--
Total acid number, mg KOH/g	664	--	0.64	1.08
Water content, wt%	1744	0.01	0.01	11.4
Sulfur by XRF, wt%	--	0.5	0.35	--
Carbon, wt%	--	--	86.91	76.02
Hydrogen, wt%	--	--	12.64	12.37
Nitrogen, wt%	--	--	--	0.306
Aromatics by HPLC, wt%	--	--	33.4	--
Aromatic ring carbon by UV, wt%				
mononuclear	--	--	9.7	--
dinuclear	--	--	10.6	--
trinuclear	--	--	0.6	--
total	--	--	20.9	--
Heat of combustion, gross, Btu/lb	240	--	19,485	17,270
Heat of combustion, gross, MJ/kg	240	--	45.32	40.18
Heat of combustion, net, Btu/lb	240	--	18,330	16,144
Heat of combustion, net, MJ/kg	240	--	42.64	37.55
Cetane Number	613	45	43	36
Distillation, °C	86			
IBP	--	--	186	--
5% evap.	--	--	210	--
10% evap.	--	--	220	--
15% evap.	--	--	228	--
20% evap.	--	--	233	--
30% evap.	--	--	241	--
40% evap.	--	--	251	--
50% evap.	--	--	258	--
60% evap.	--	--	267	--
70% evap.	--	--	276	--
80% evap.	--	--	287	--
90% evap.	--	338	302	--
95% evap.	--	--	320	--
EP	--	371	338	--
Recovered, %	--	--	98.5	--
Residue, %	--	--	1.5	--

\* FRF composition: 10135: 84.7 vol%  
EA-96: 5.3 vol%  
Water (180 ppm TDS): 10.0 vol%

\*\* Pilot flame extinguished above 74°C, no flash point below 100°C

\*\*\* Kinematic viscosity at 38.8°C



APPENDIX C

EMULSIFYING AGENTS REFERENCED IN THIS REPORT

TABLE C-1. EMULSIFYING AGENTS REFERENCED IN THIS REPORT

<u>EA No.</u>	<u>Code No.</u>	<u>Manufacturer</u>	<u>Mfg's Batch No.</u>	<u>Mfg's I.D.</u>	<u>TAN*, mg KOH/g</u>
90	1000-2	Clintwood	6906	LT-19-21-2	19.0
96	10360	Clintwood	6905	LT-19-21-2	19.0
99	10484	Clintwood	7081	LT-19-21-1	15.5

\* ASTM D 664



APPENDIX D  
PROPERTIES OF AROMATIC CONCENTRATES



TABLE D-1. ANALYSIS OF COMMERCIAL AROMATIC CONCENTRATES

Property	Test Method ASTM D	Aromatic Concentrate Code No.		
		10716	10748	10749
Density, g/mL at 20°C		0.8646	0.8931	0.8854
Flash point, PMMC, °C	93	39	67	43
Kinematic viscosity, cSt, at 0°C	445	--	2.01	1.90
Kinematic viscosity, cSt, at 40°C	445	0.75	--	--
Heat of combustion, net, Btu/lb	240	17560	--	--
Heat of combustion, net, MJ/kg	240	40.85	--	--
Aromatic ring carbon by UV, wt%				
monocyclic	--	52.46	49.57	52.24
dicyclic	--	1.65	6.50	4.97
tricyclic	--	0.00	0.16	0.05
total	--	54.11	56.23	57.26
Sulfur by XRF, wt%	--	0.00	--	--
Carbon, wt%	3178	89.23	--	--
Hydrogen, wt%	3178	10.07	--	--
Cetane number	613	5.0	--	--
Distillation, °C	86			
IBP		157	184	170
5% evap.		160	187	179
10% evap.		161	188	181
20% evap.		161	189	183
30% evap.		161	190	184
40% evap.		162	191	186
50% evap.		162	191	188
60% evap.		163	192	189
70% evap.		164	193	192
80% evap.		167	196	196
90% evap.		169	200	202
95% evap.		172	204	208
EP		194	231	234
Recovered, %		99.0	99.0	99.0
Residue, %		1.0	0.5	0.5
Loss		0.0	0.5	0.5

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APPENDIX E

TYPES OF ENGINES AND FUEL FILTERS AND FUEL TANK CAPACITIES  
IN VEHICULAR FUEL SYSTEMS



TABLE E-1. ENGINES, FUEL FILTERS AND TANK CAPACITY  
OF SOME DIESEL-POWERED VEHICLES\*

Engine	Fuel Filters		Vehicle (Tank Capacity in Gallons)
	Primary	Secondary	
AVDS-1790-2A	FRAM 8395476	TACOM 570269D	M48A3(375) M60(385), M60A1(375), M60A2(385), M728(515)
AVDS-1790-2C	Bendix 11668617	-	M60A2(385) M88(445)
AVDS-1790-2D	Bendix	-	M60A1(375)
AVDS-1790-2D	11668617		M60A2(385) M728(525)
DD 6V-53	AC 557032	AC 5574508	M106A1(95) M113A1(80) M125(95), M132A1(95) M577A1(120), M667(85) XM727(190), M730(190)
DD 8V-71T	AC 557032	AC 5574508	M108(50), M109(50) M109A1(50), M109A3(50) M578(320)
ENDT-673	MAC 237GB16	MAC 237GB13	M51A1(90), M52A1(110) M54A1(78), M54A1C(78) M55A1(66)
LD-465-1 LD-465-1C LDT-465-1C	TACOM 11610298	TACOM 11610298	M35A2(50), M35A2C(50) M36A2(50), M44A2(50) M45A2(50), M46A2(50) M46A2C(50), M49A2C(50) M50A2(50), M109A2(50) M185A3(50), M275A2(50) M342A2(50)
LDS-427-2	TACOM 11609954	TACOM 11609954	M35A1(50) M49A1C(50) M109A2(50) M275A1(50)
LDS-465-1 LDS-465-1A	Purolator 33435	TACOM 11610298	M40A2C(110) M51A2(90) M54A2(78) M55A2(66) M63A2(66) M63A2C(66) M246A2(133) M291A2(66) M291A2C(66) M543A2(110)
LDS-465-2	TACOM 11610298	TACOM 11610298	M656(133)
NHC-250	Cummins Filter Water-SEP 256-546	Cummins "In Pump" 146483	M813(78), M814(78) M816(133), M817(110) M819(78), M821(78) M818(110)

\* Source: Reference 7 and U.S. Army Tech Manual TM43-0001-31

APPENDIX F  
DEFINITIONS, ABBREVIATIONS, AND ACRONYMS

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## DEFINITIONS, ABBREVIATIONS, AND ACRONYMS

### Definitions:

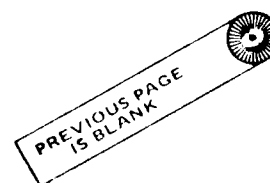
Ca (NO <sub>3</sub> ) <sub>2</sub>	- Calcium nitrate
DF-A	- VV-F-800B-DF-A. Arctic-grade diesel fuel
DF-1	- VV-F-800B-DF-1: Winter-grade diesel fuel
DF-2	- VV-F-800B-DF-2: Diesel fuel
F-54	- NATO diesel fuel
JP-4	- Gasoline-type military jet fuel
JP-8	- Kerosine-type military jet fuel
KOH	- Potassium hydroxide
NaCl	- Sodium chloride

### Acronyms:

AC	- Aromatic concentrate (C <sub>9+</sub> aromatics)
AFLRL	- U.S. Army Fuels and Lubricants Research Laboratory
ASTM	- American Society for Testing and Materials
EA	- Emulsifying agent
FIA	- Fluorescence indicator adsorption (ASTM D 1319)
FRF	- Fire-resistant diesel fuel
HPLC	- High-performance liquid chromatography
LOA	- Letter of Agreement
NATO	- North Atlantic Treaty Organization
OA	- Oleic acid
PMCC	- Pensky-Martens closed cup flash point test (ASTM D 93)
TAN	- Total acid number (ASTM D 664)
TDS	- Total dissolved solids
TARC	- Total aromatic ring carbon content
UV	- Ultraviolet absorption spectroscopy
XRF	- X-ray fluorescence analysis

### Engine Designations:

AVDS-1790-2A, B, C, or D  
DD 4-53T  
DD 6V-53T  
DD 8V-71T  
(SPEC22.A)





Engine Designations (Cont'd)

ENDT-673

LD 465-1 or 1C

LDT 465-1C

LDS 427-2

LDS 465-1, 1A, or 2

NHC-250

Vehicle Designations:

M113A1 - Armored personnel carrier

M60A3 - Battle tank

Filter Brands:

AC

FRAM

Bendix

MAC

TACOM

Cummins

LOA Acronyms:

C-130 - Military transport aircraft

C-141 - Military transport aircraft

C-5A - Military transport aircraft

APC - Armored personnel carrier

MOS - Military Occupational Specialty

RAM - Reliability and maintainability

DT-I - Developmental Test-I

DT/OT-I - Developmental Test/Operational Test-I

OT-I - Operational Test-I

VAL IPR - Validate In Process Review

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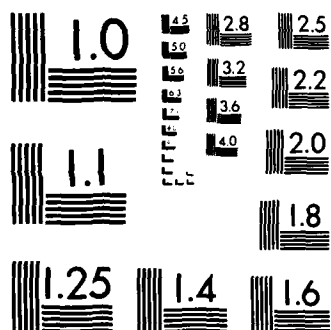
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